National Research Centre" Kurchatov Institute"
European Organization for Nuclear Research

Investigations of fast proton irradiation influence using NRC KI cyclotron on superconductor materials for Large Hadron Collider magnets

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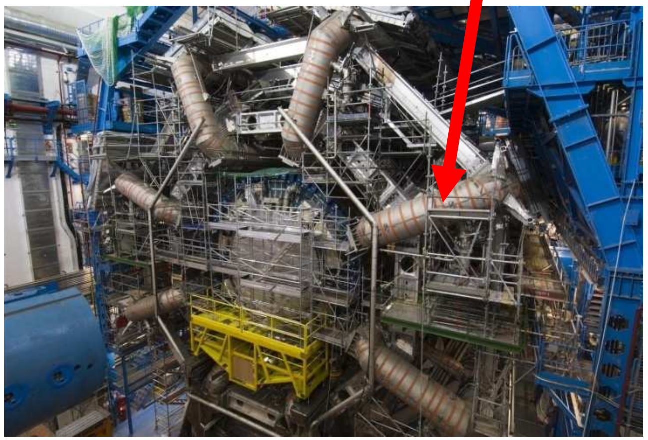
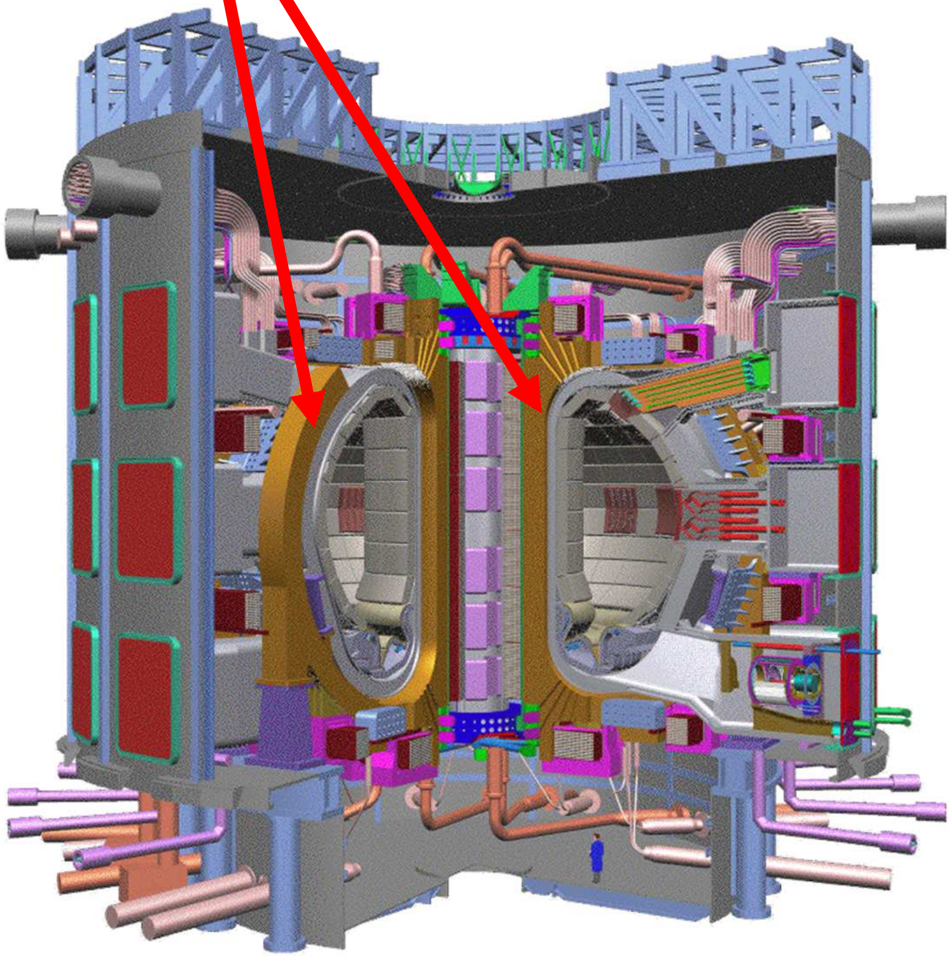
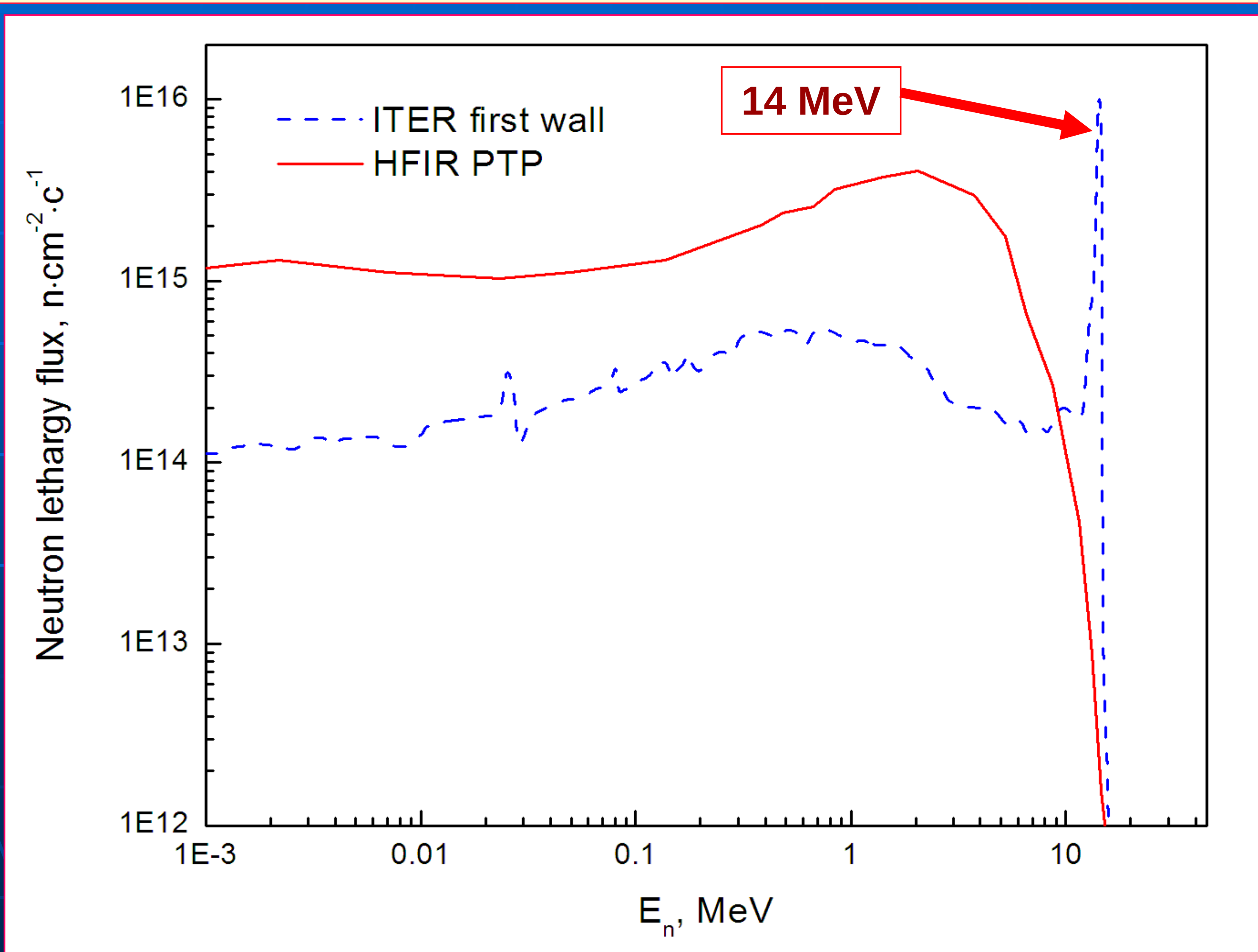
Superconductor magnets for LHC and ITER



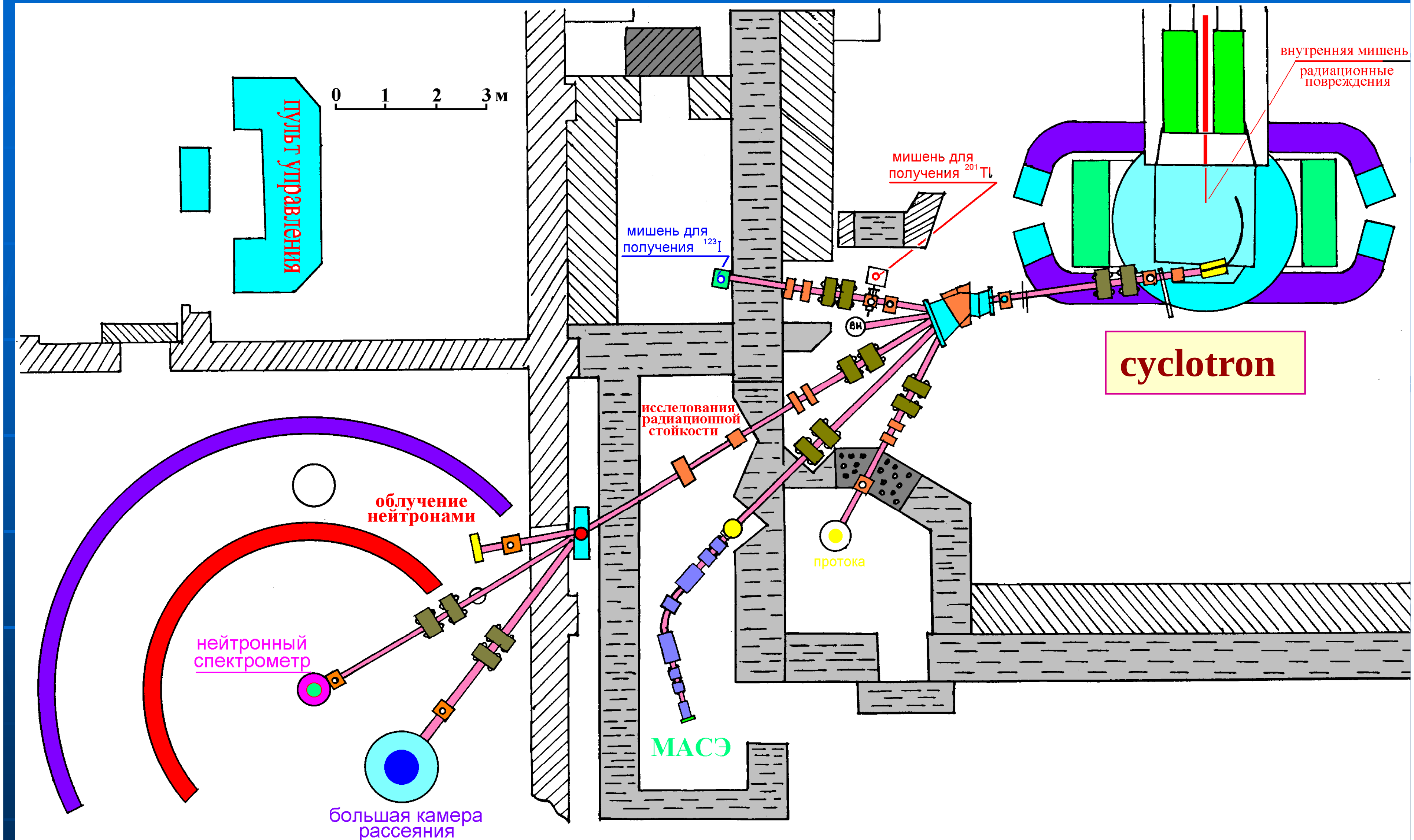


Neutron Energy Fluxes for different Fast Neutron Facilities (HRIR, ITER)



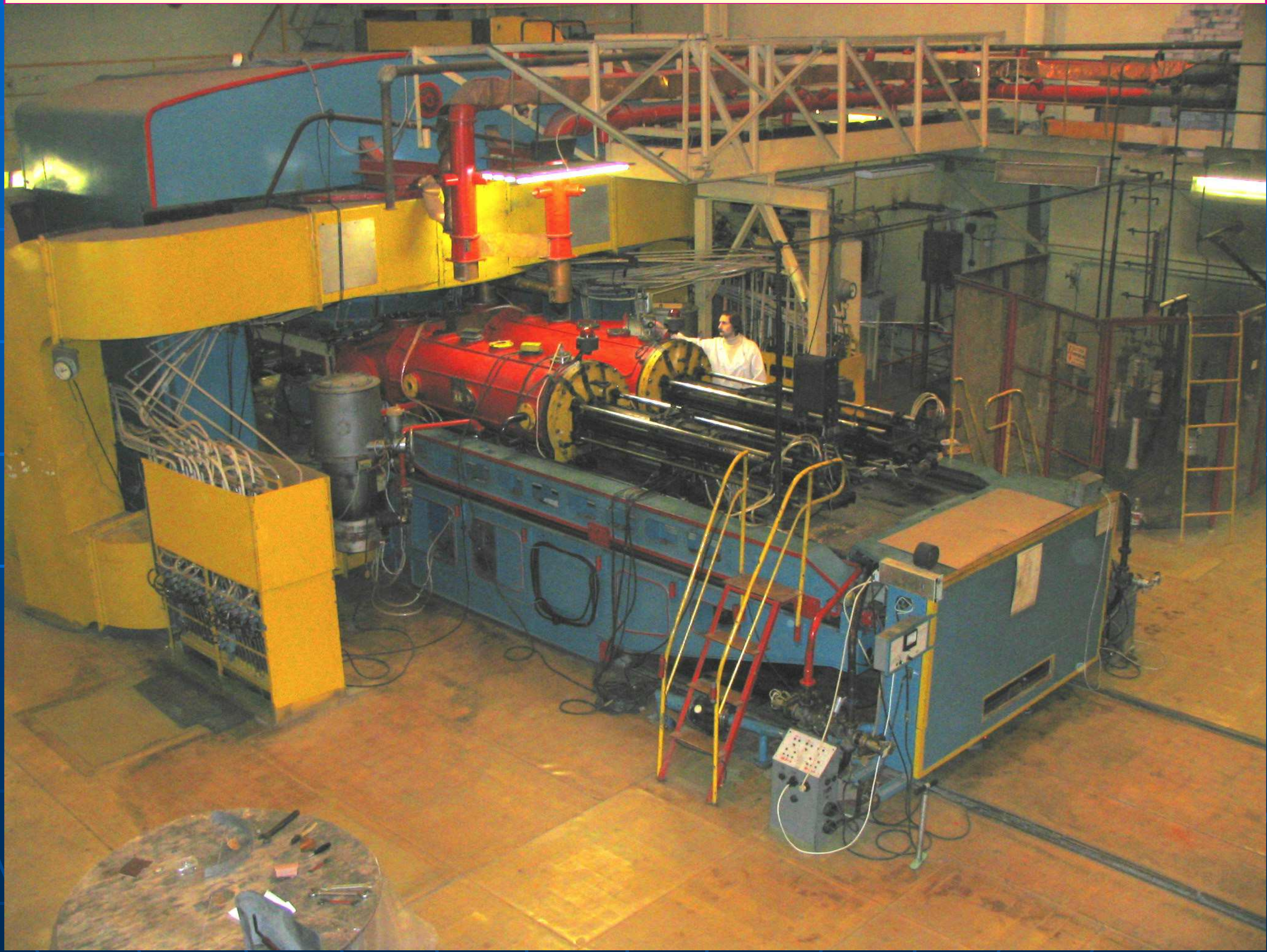
R&D program objectives

- Examine the sensitivity of new materials (High-J_C, optimized ternary Nb_3Sn, MgB_2 and HTS) and stabilizer (Cu) to LHC radiation (neutrons **and** protons) with distributions peaked at:
  - **1 MeV neutrons**
  - **60 MeV protons**
  - Significant tails at higher energy
- This is a ***new domain*** **for which very little and very scattered data exists**



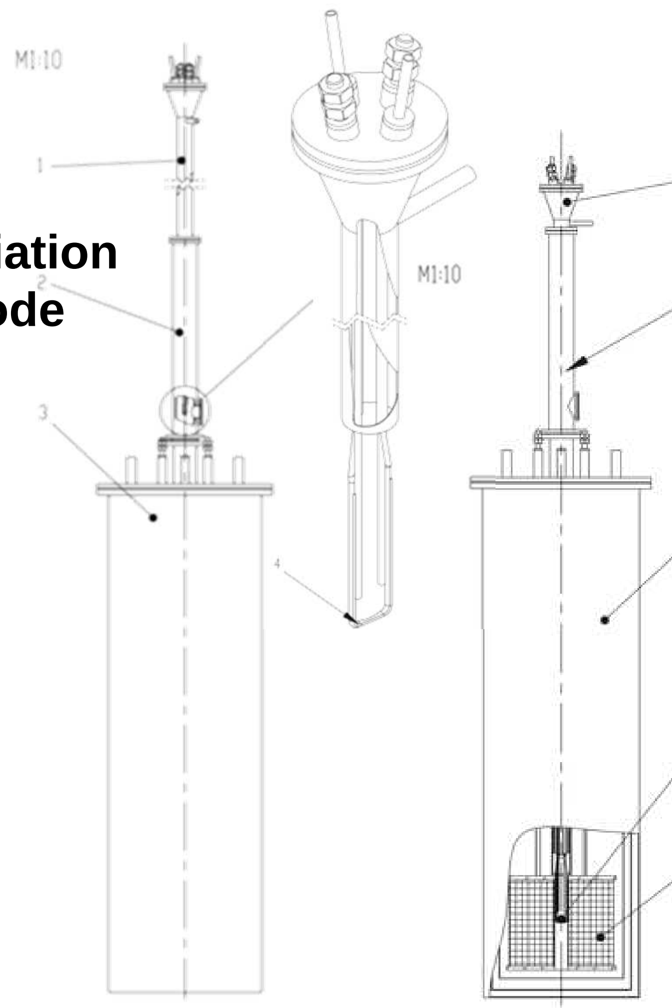
System of cyclotron transportation

Cyclotron of RRC “Kurchatov Institute”



Kurchatov Institute - Moscow

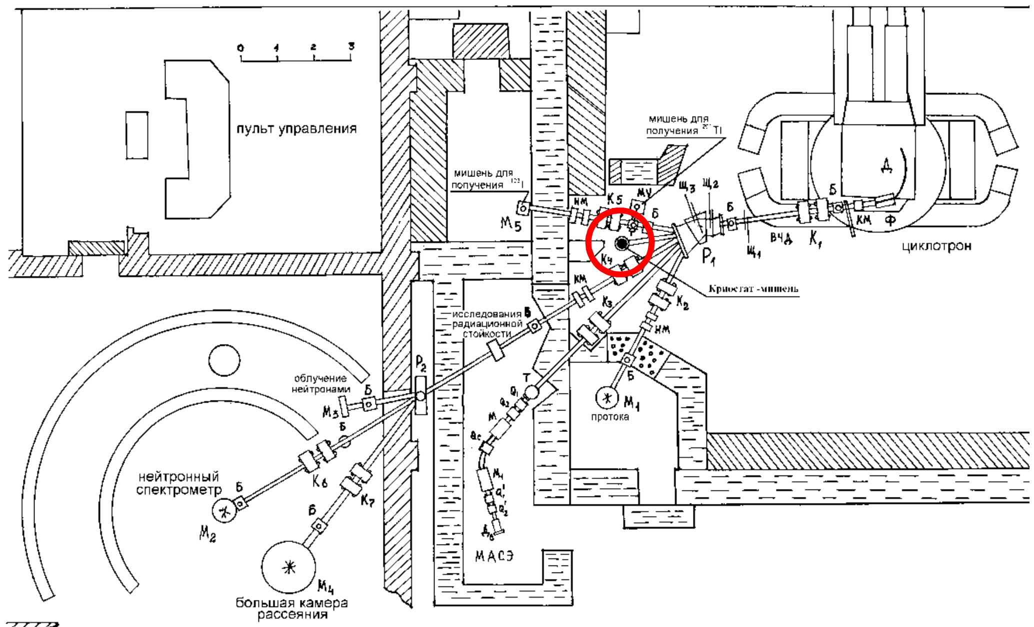
Irradiation mode



Measurement mode

12T, 2 kA I_C test station

35 MeV cyclotron



система транспортировки циклотрона

Accelerators of Charge Particles of National Research Centre “Kurchatov Institute”

Cyclotron of NRC KI:

protons with energy < 35 MeV, current J < 30 mkA

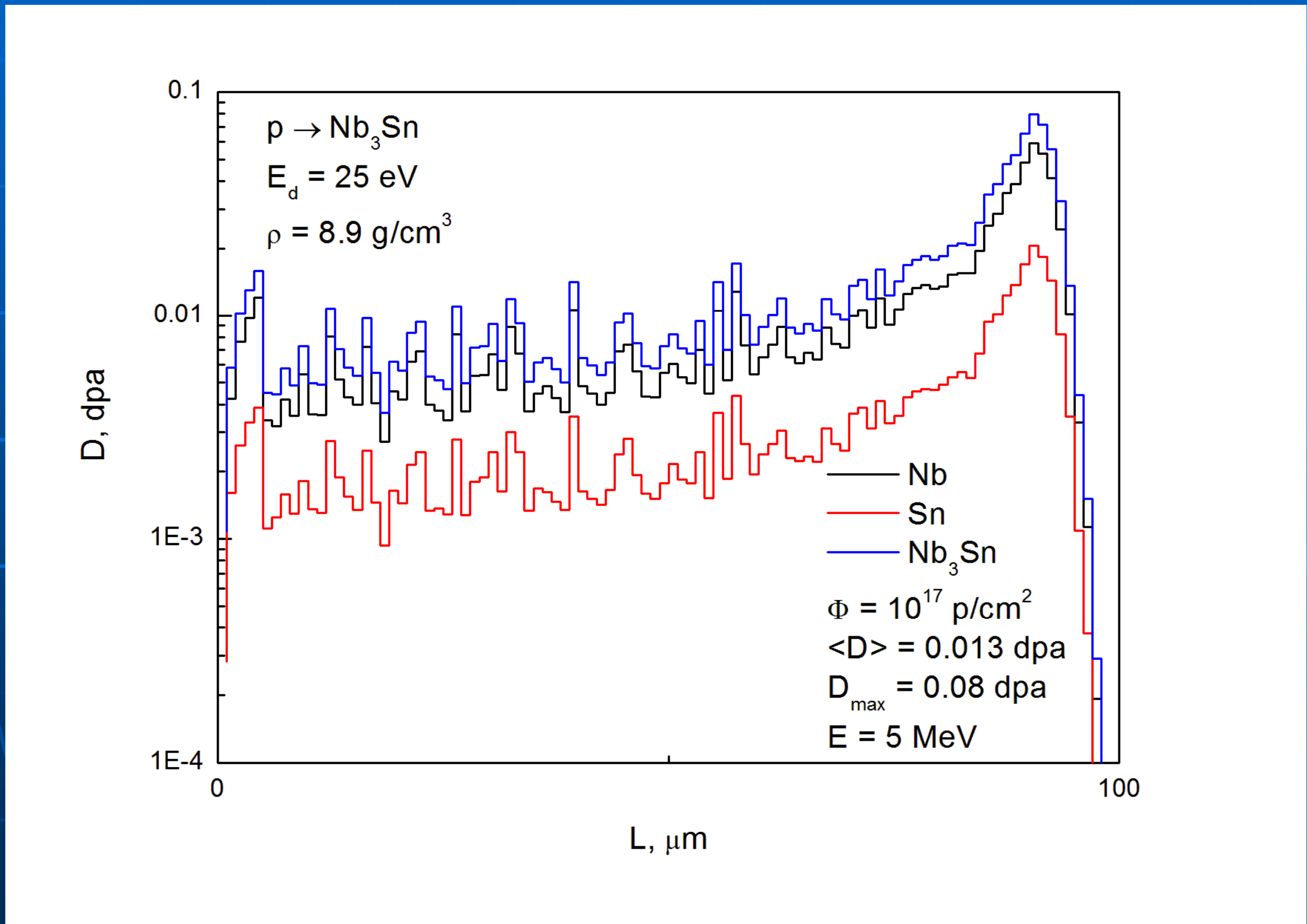
helium ions He^4 with energy < 60 MeV, current J < 20 mkA

ions O^{16} with energy < 120 MeV , current J < 5 mkA

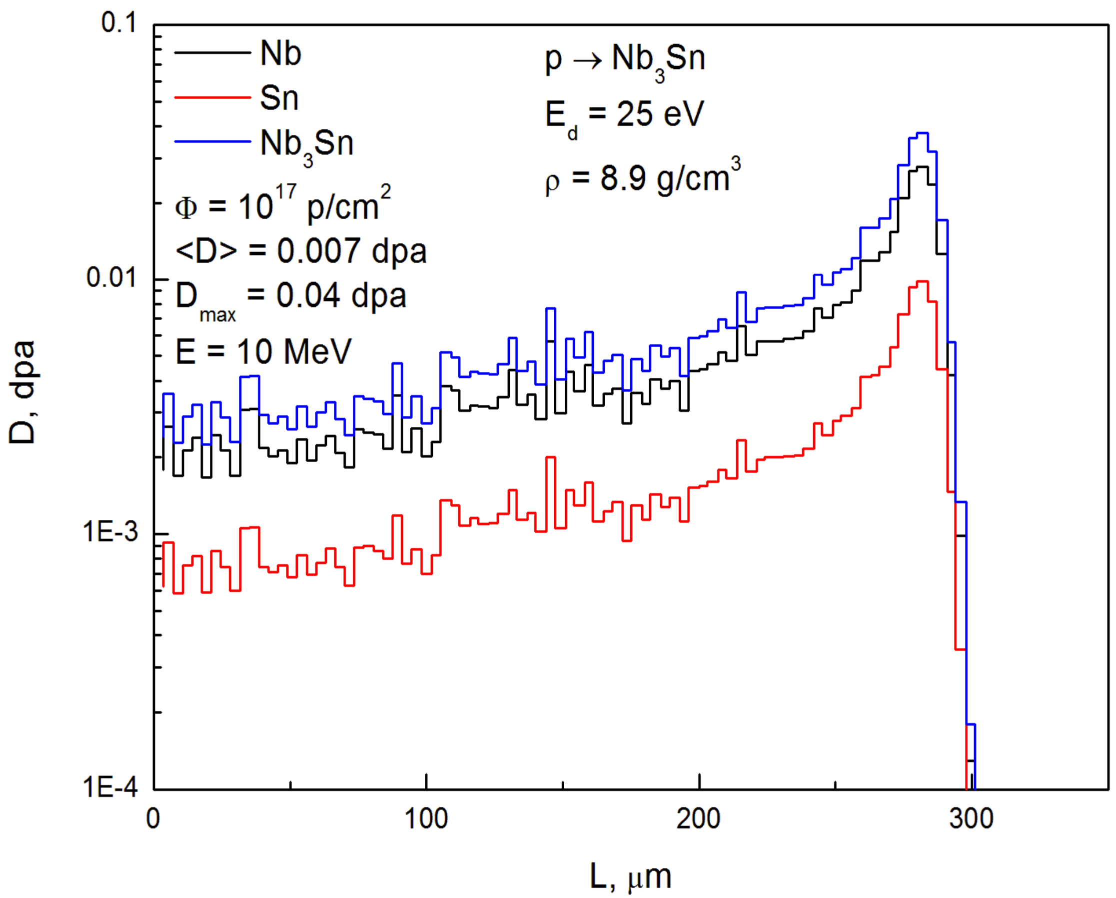
ions C^{12} with energy < 80 MeV, current J < 5 mkA

Theoretical calculations of radiation damage profiles at different fast particle energies for irradiation of Nb_3Sn samples on NRC KI cyclotron

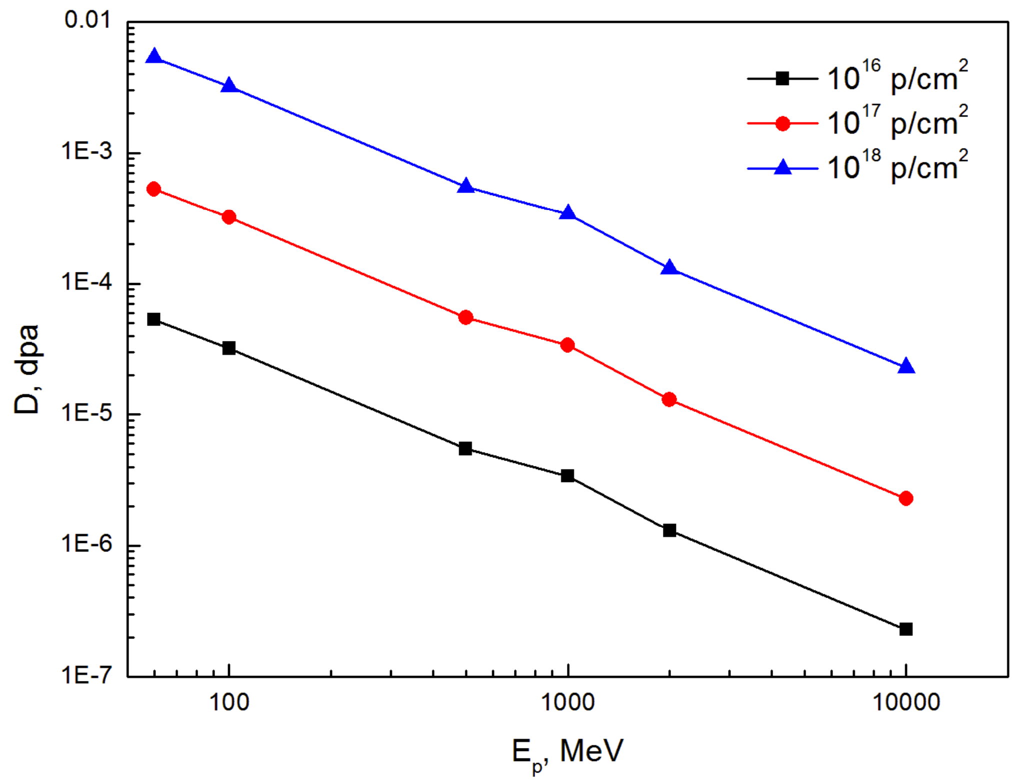
Radiation damage profile in Nb3Sn under 5 MeV proton beam irradiation on NRC KI cyclotron



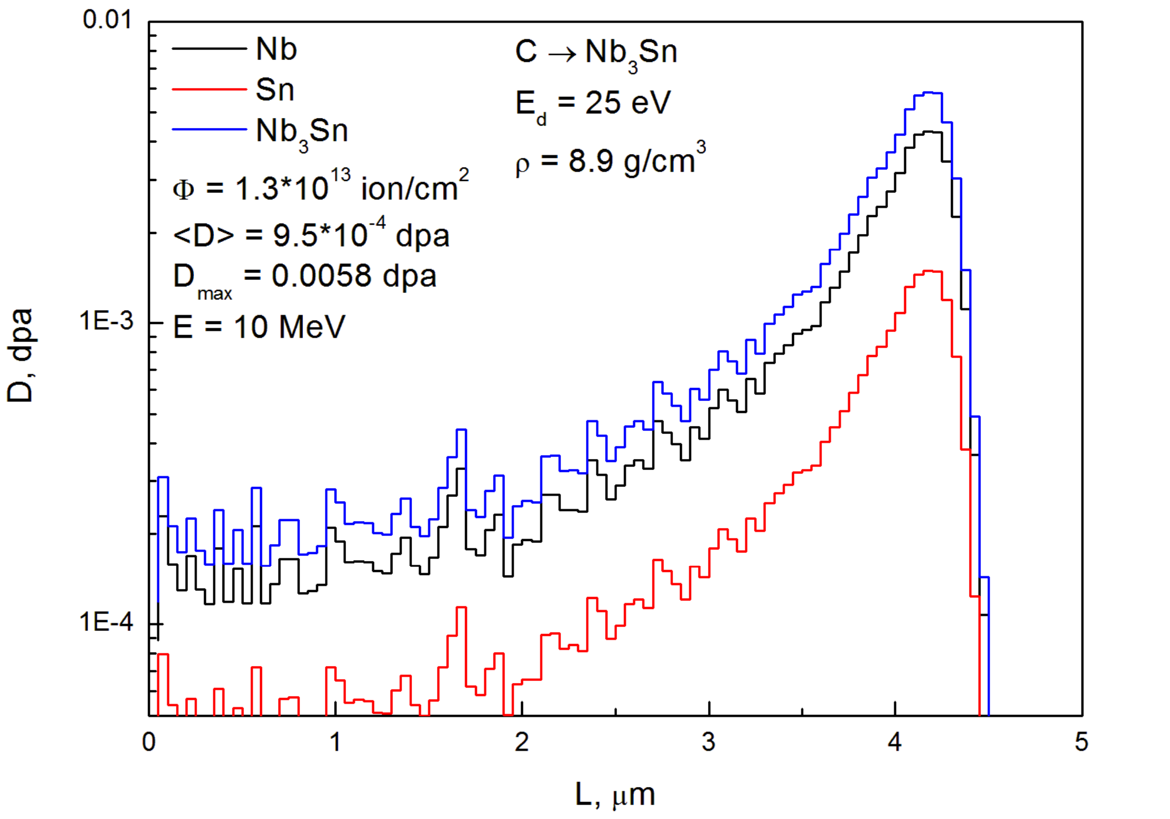
Radiation damage profile in Nb_3Sn under 10 MeV proton beam irradiation on NRC KI cyclotron



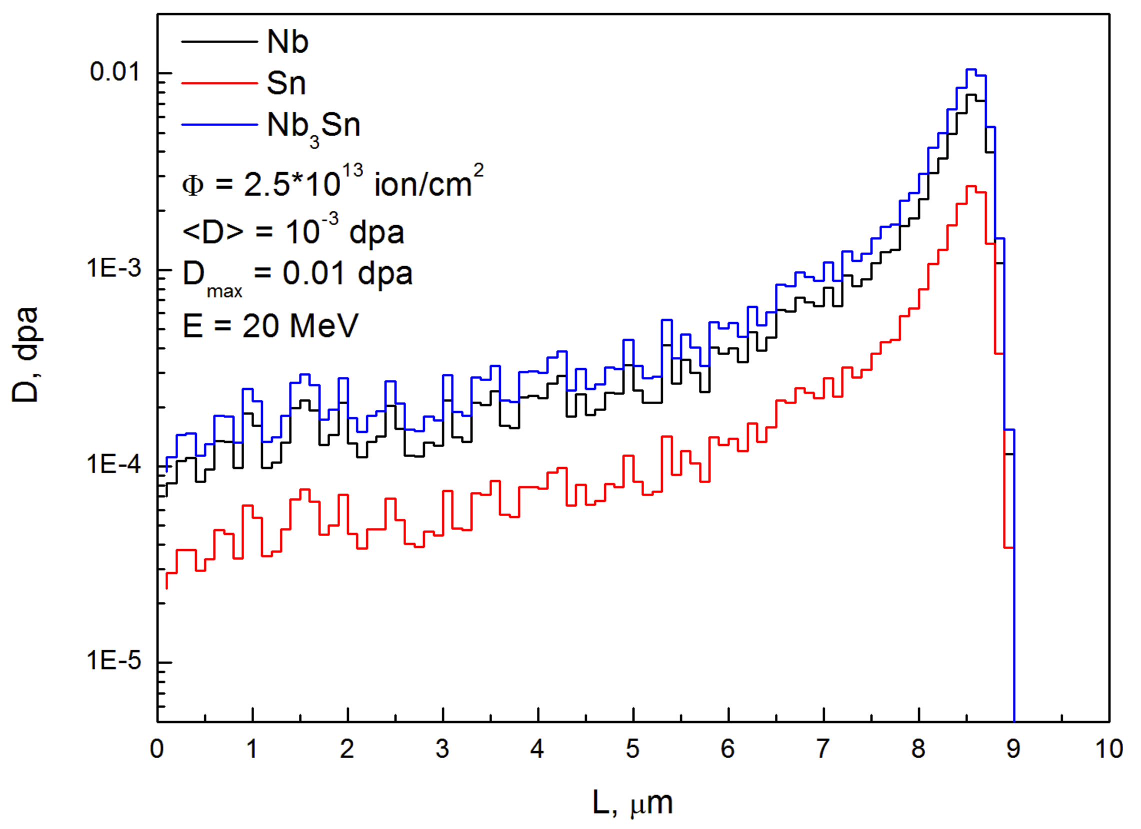
Radiation damage profiles in Nb_3Sn under different proton beam energy irradiation on NRC KI cyclotron



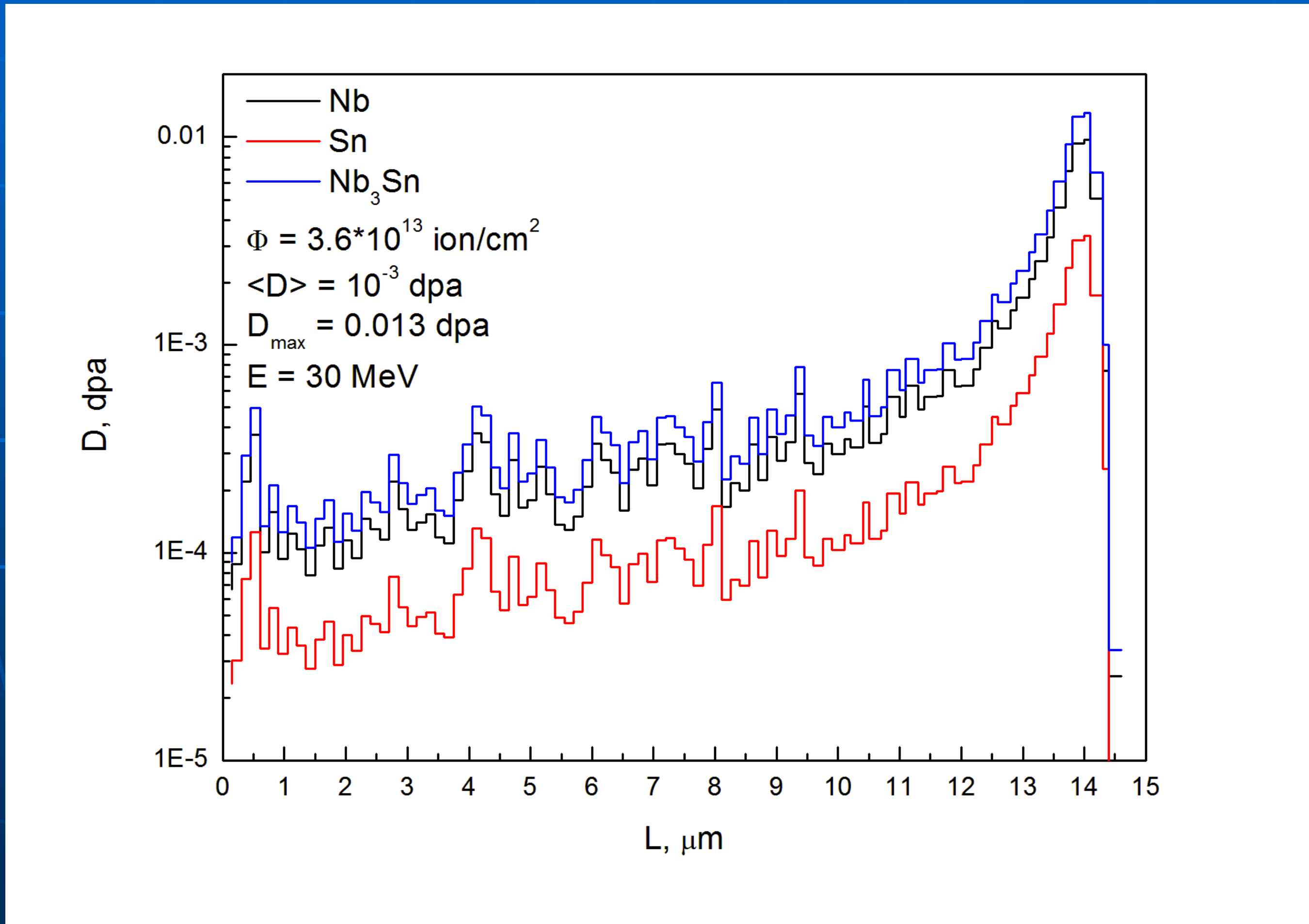
Radiation damage profile in Nb_3Sn under 10 MeV carbon beam irradiation on NRC KI cyclotron



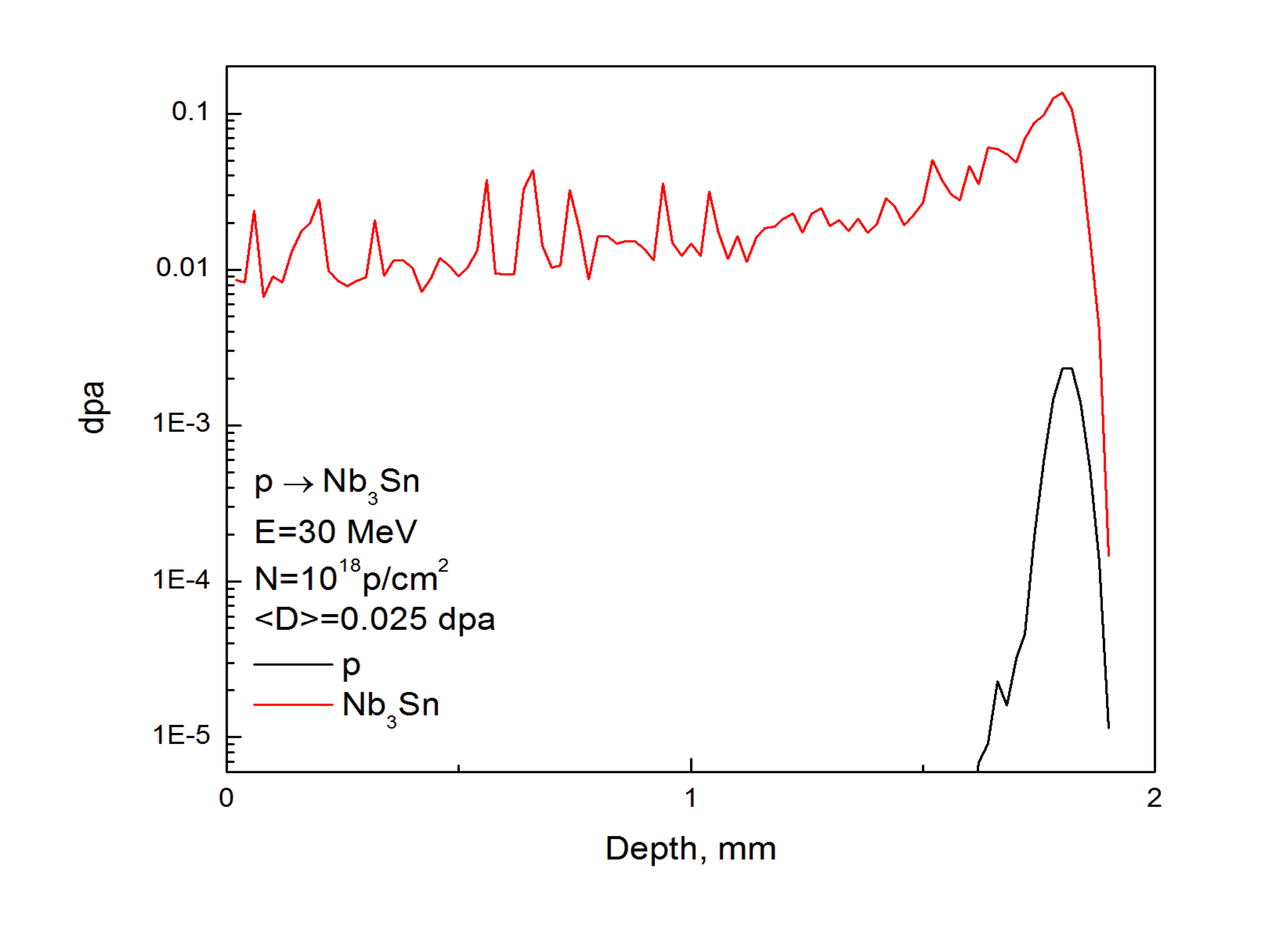
Radiation damage profile in Nb_3Sn under 20 MeV carbon beam irradiation on NRC KI cyclotron



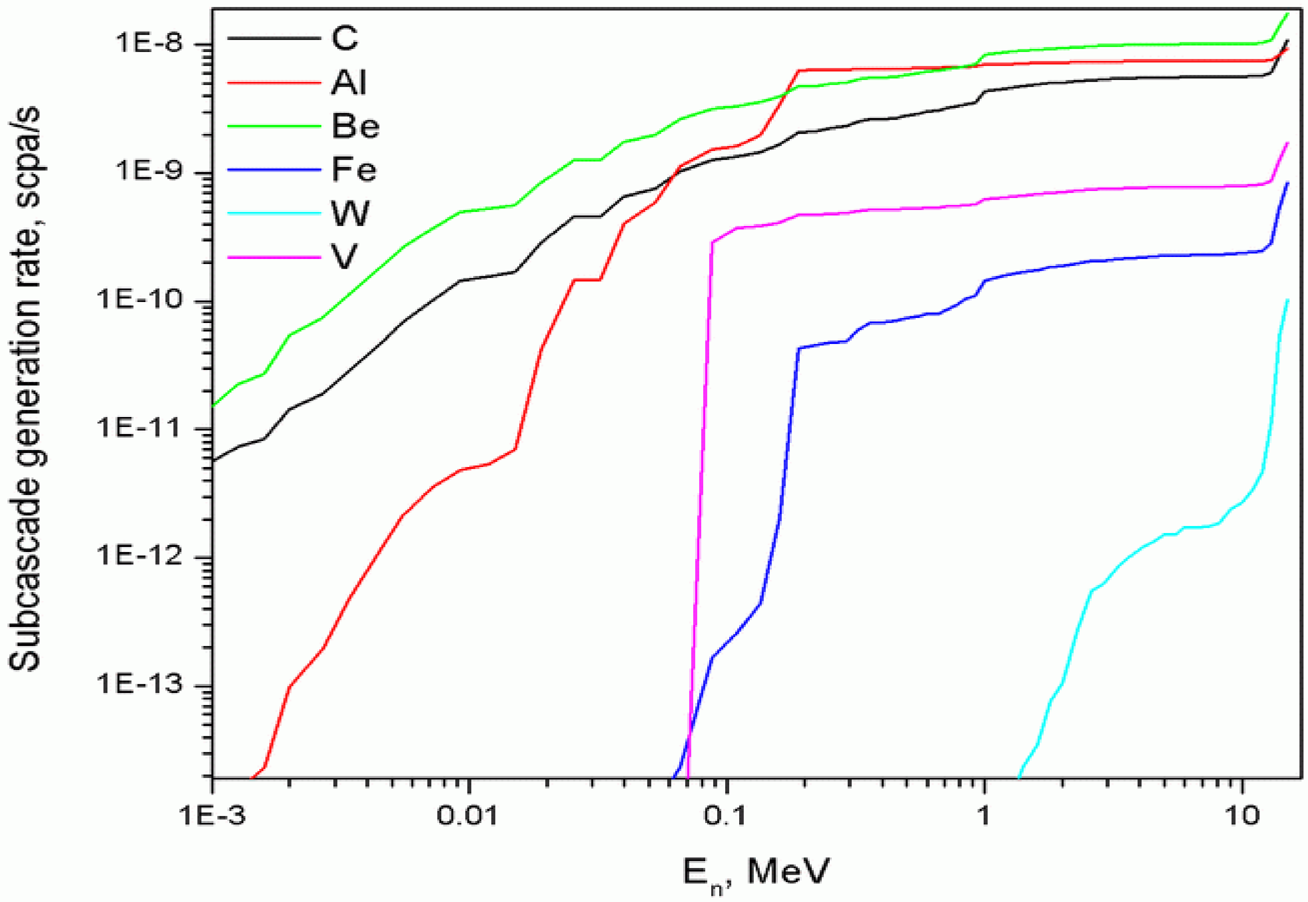
Radiation damage profile in Nb_3Sn under 30 MeV carbon beam irradiation on NRC KI cyclotron



Radiation damage profile in Nb_3Sn under 30 MeV proton beam irradiation on NRC KI cyclotron



Sub-cascade Generation Rate in different Materials under Neutron Irradiation in DEMO



Critical current measurements of Nb_3Sn (initial samples) at NRC KI

Critical current measurements were carried out in Kurchatov Institute facility in magnetic field up to 12T.

Two kind of U-shape holders for measurement were used:

- **Sample 1:** **-"Vanadium holder"**
- **– 15 cm x 3 cm U-shape with section 5 mm x 1 mm. The material is V(Cr3%at+Ti3%at.) alloy. Thick layer of copper (about 50 mkm thick) were plated on the flat surface using plasma-gas method. Reacted sample #0802 was soldered using lead-tin solder (Fig.1a and 1b). Unfortunately during formation of sample holder copper layer was peeled of vanadium holder on external corners (see Fig.1a) possibly due to high thickness of copper layer. Additional stainless steel support was used to fix firmly Nb3Sn wire (see Fig.1b).**
- **Sample 2:** **-"Stainless steel holder"**
- **- 15 cm x 3 cm U-shape with section 5 mm x 1 mm. Reacted sample #0802 was soldered using lead-tin solder with orthophosphoric acid flux.**
- **Each sample was mounted on standard inset for critical current measurement designed for currents range up to 1.5 kAmps.**

Sample 1 (“Vanadium holder”) – holder forming


Fig.1a

Sample 1 (“Vanadium holder”) – soldered sample

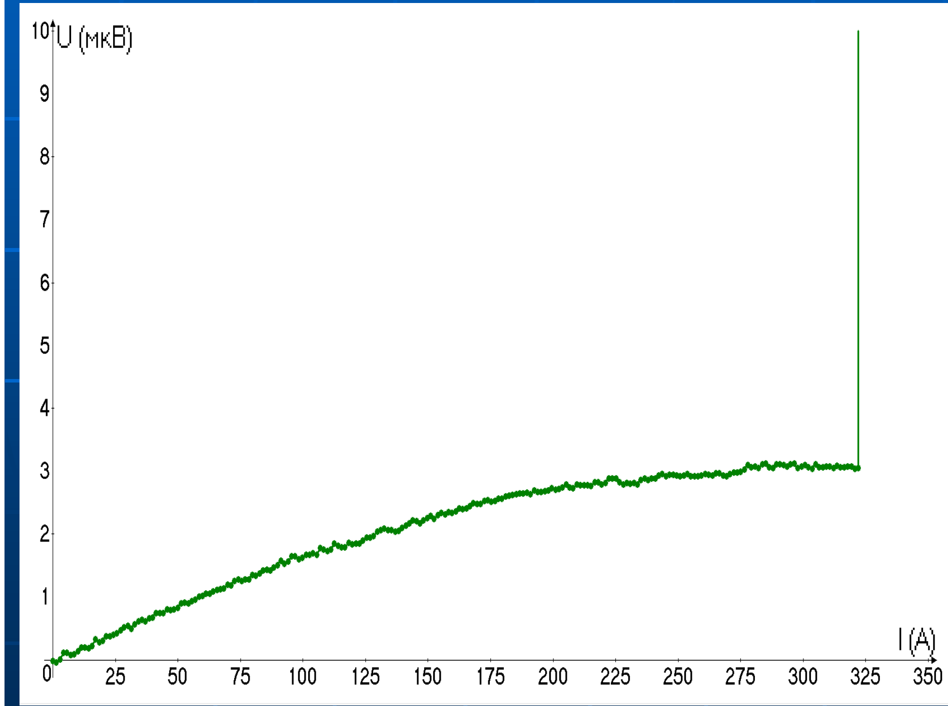
Measurements

- Critical currents were measured used automatized facility equipped with Keithley`s nanovoltmeters and Ammeter Sorensen SGI Series current sources (1200 A x 10V each, can be combiner in parallel). Cryostat with magnetic field 13 T with bore 40 mm was used.
- Both nano-volt signal from 1 cm of sample and from sample holder (protection signal on 33 cm) were registered simultaneously. Temperature was about 4.2 K (liquid helium). No special temperature corrections were done. Measurements were carried out on 26 Apr 2013.

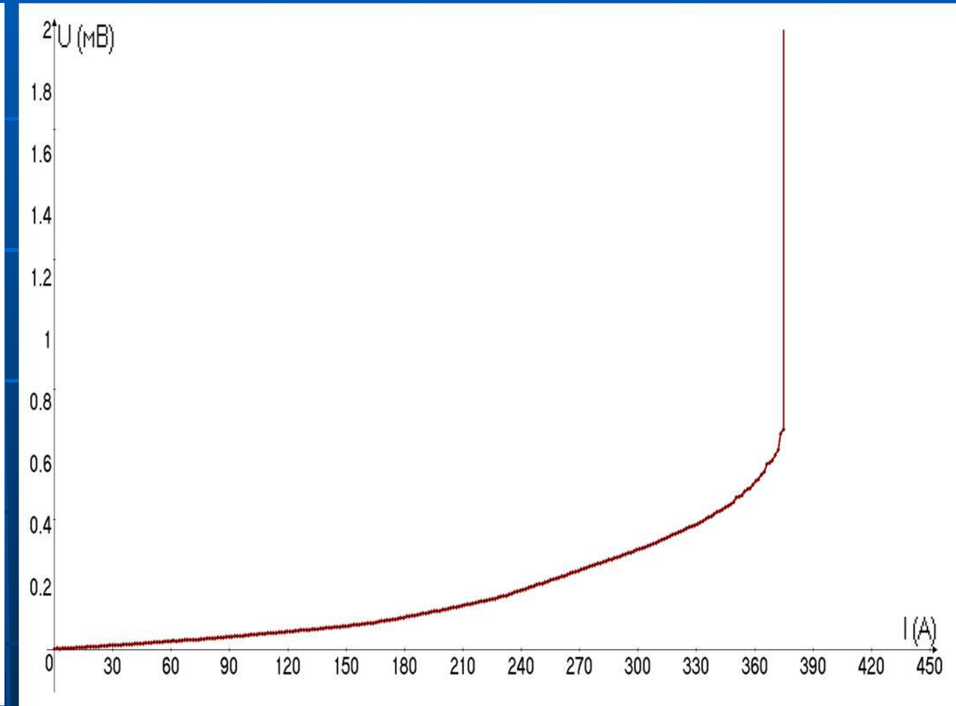
The results for volt-ampere characteristics of Nb3Sn billet # 0802 samples

Sample #1.Vanadium holder. Nb3Sn billet #0802. Signal Distance L=11 mm.

1) Magnetic Field 9 T



VACh of sample signal

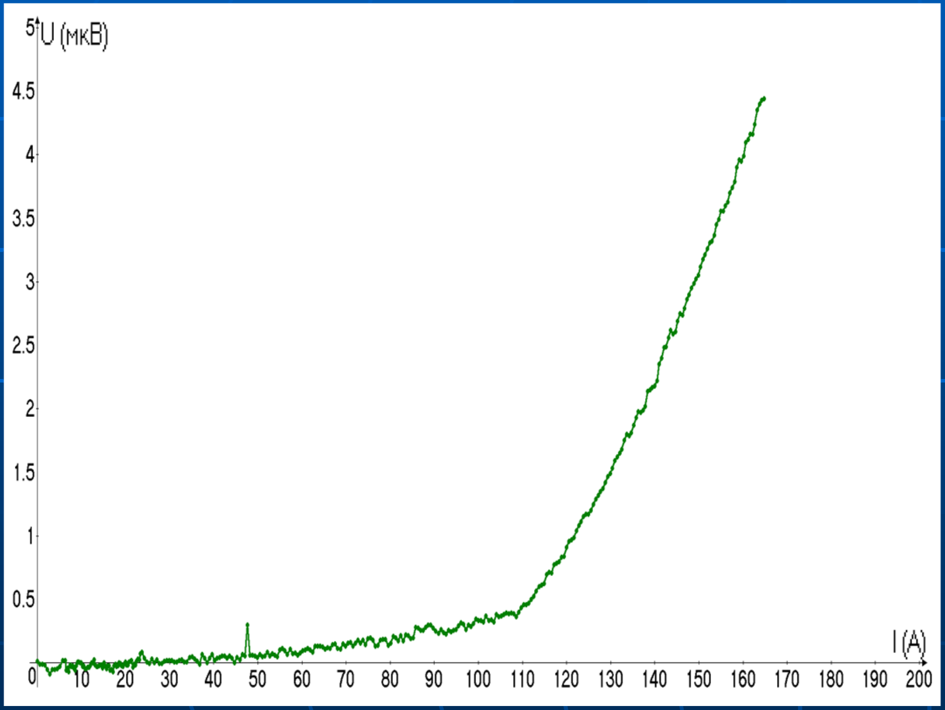


VACh of protection signal

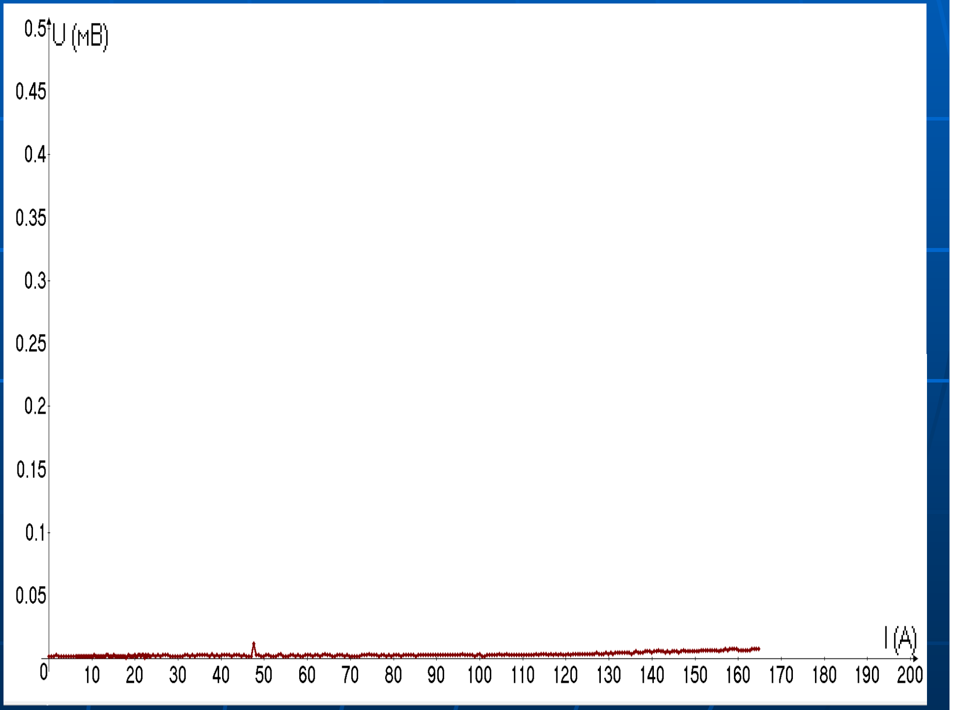
The results for volt-ampere characteristics of Nb3Sn billet # 0802 samples

Sample #2. Stainless holder. Nb3Sn billet # 0802. Signal Distance L=12 mm.

1) Magnetic Field 9 T



VACh of sample signal



VACh of protection signal

Discussion

1. Only Nb3Sn billet #0802 pilot experiment was done on samples mounted on two various U-shape holders.
2. Both samples demonstrated insufficient results.
3. The Sample #1 (Vanadium holder) demonstrated voltage surge due to bad connection to holder (probably, see raw VACh`s above).
4. The Sample #2 (Stainless steel holder) in contrast demonstrated good holder signal, but very small measured critical current. Possible this is due to damage of central part of sample.

Magnetization measurements of Nb_3Sn (initial samples)

Sample preparation

- **Samples for measurements were cut from Nb3Sn (24.8% Sn) foil MST-2 with <u>thickness of 220 mkm</u>. Sizes of samples were close to square form:**

  1) <u>Sample1: 2 x 2 mm</u>. Mass m1= 6.2 mg

  2) <u>Sample2: 2.5 x 2.2 mm</u>. Mass m2= 9.7 mg

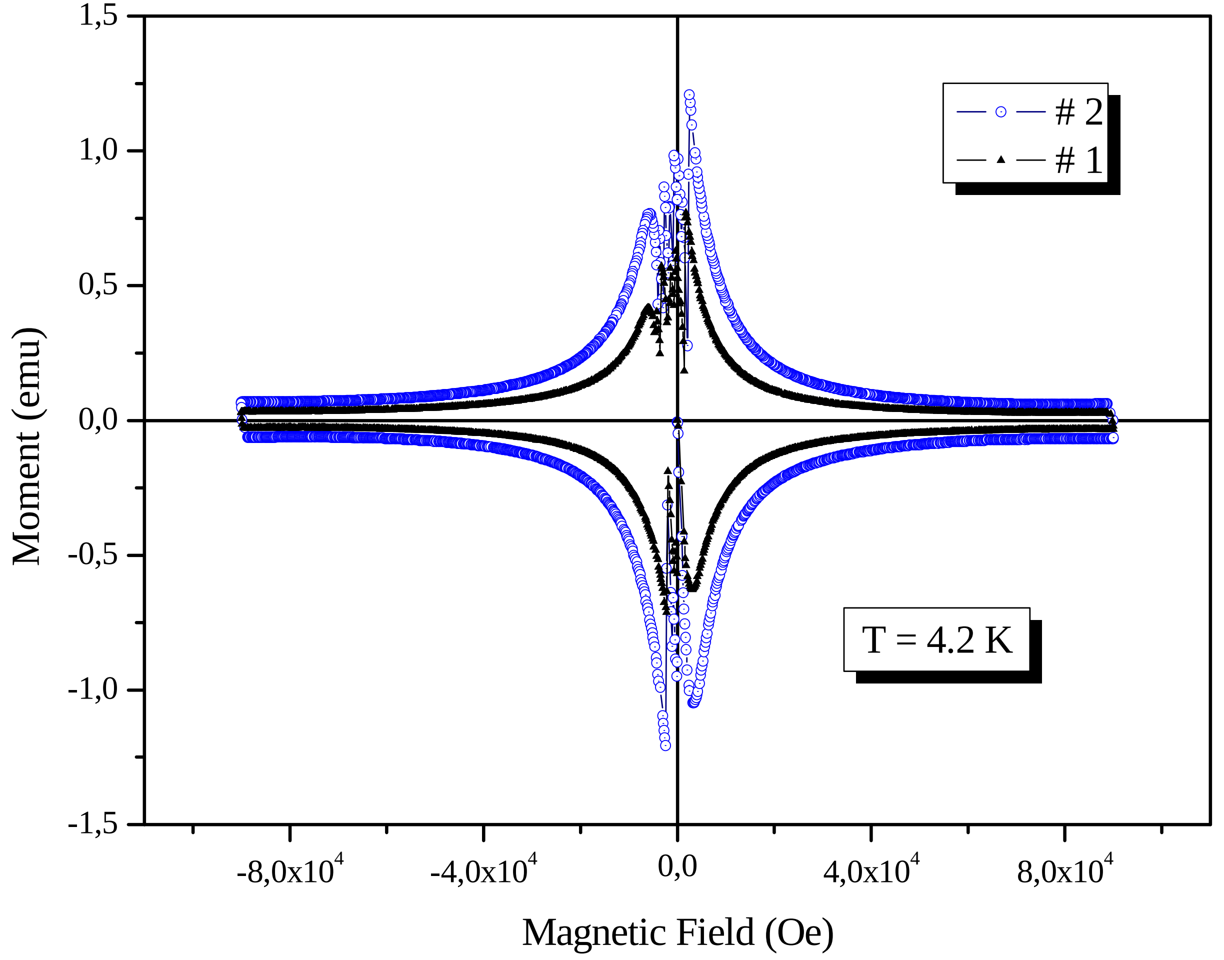
- **Each sample was mounted on standard sample holder in orientation of external magnetic field to be perpendicular to sample plane.**

Measurements

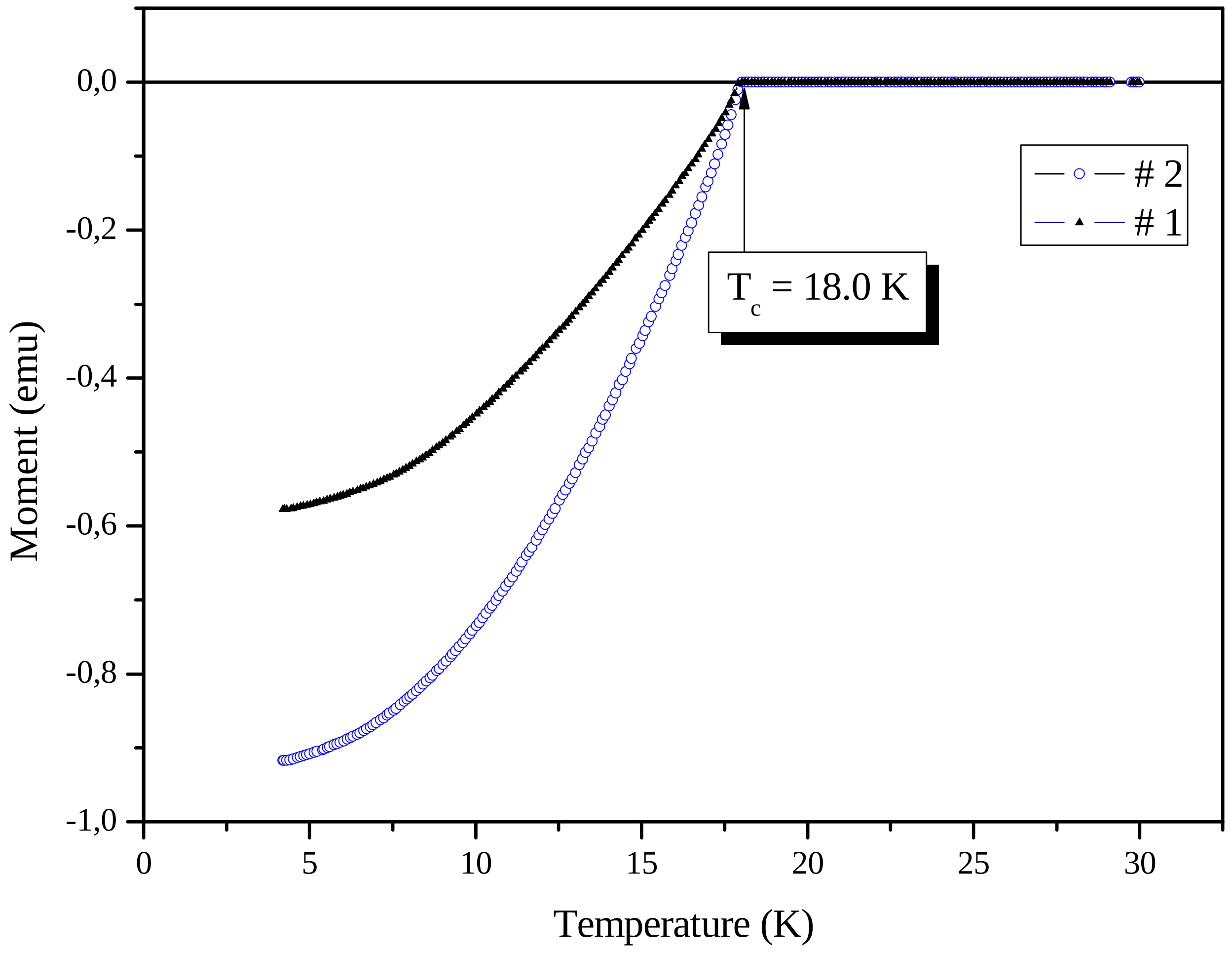
- Each sample was cooled in “zero magnetic field” (residual field was about 10 Oe) down to temperature of 4.2 K. Then magnetization curves M(H) were registered with field sweep rate of 150 Oe/sec. Field limits was ±9 T.

- After magnetization curves M(H) measurements the dependence of residual magnetic moment was measured up to temperature of T=30 K. Temperature rate was 1 K/minute (0.017 K/s).

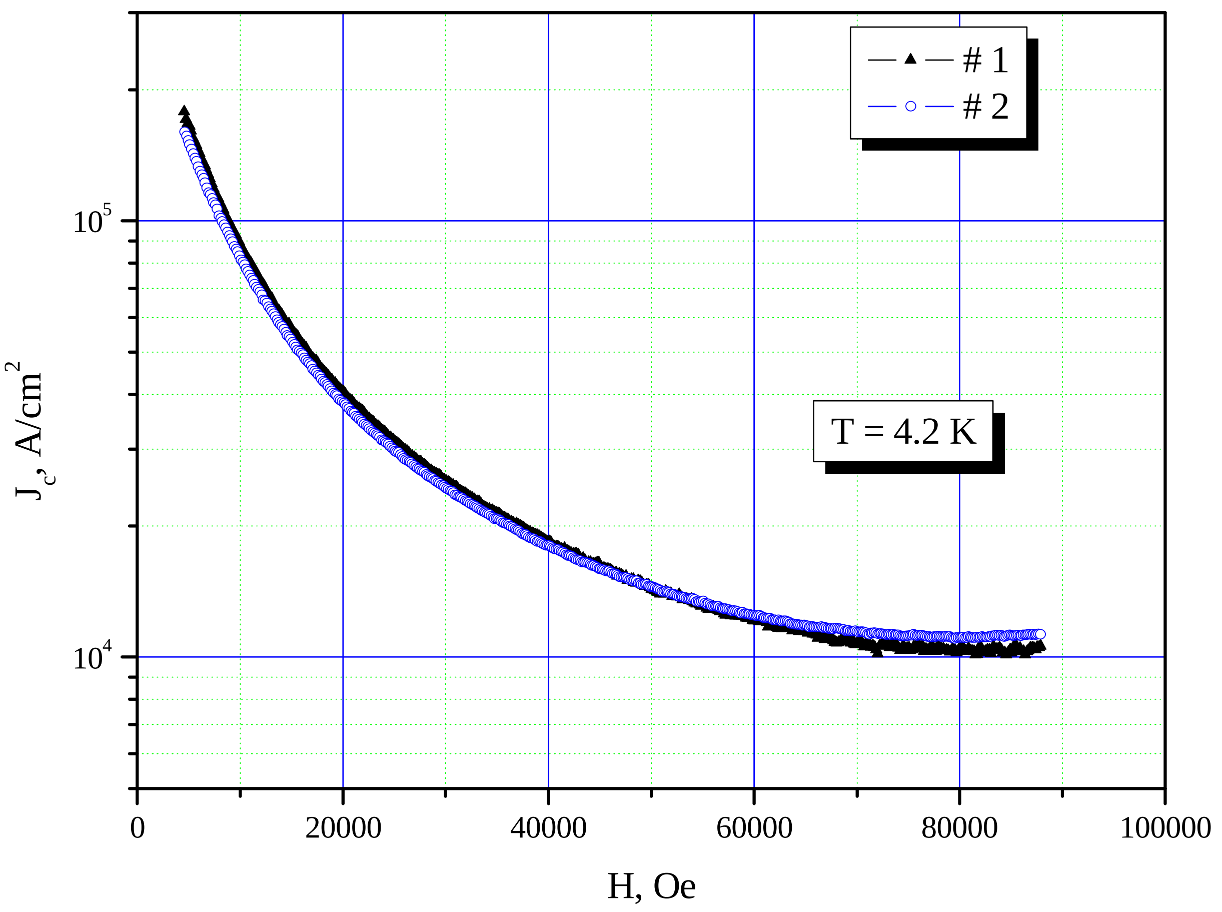
Magnetization curves M(H). Some jumps in low-field region possibly due to nonhomogeneity of samples



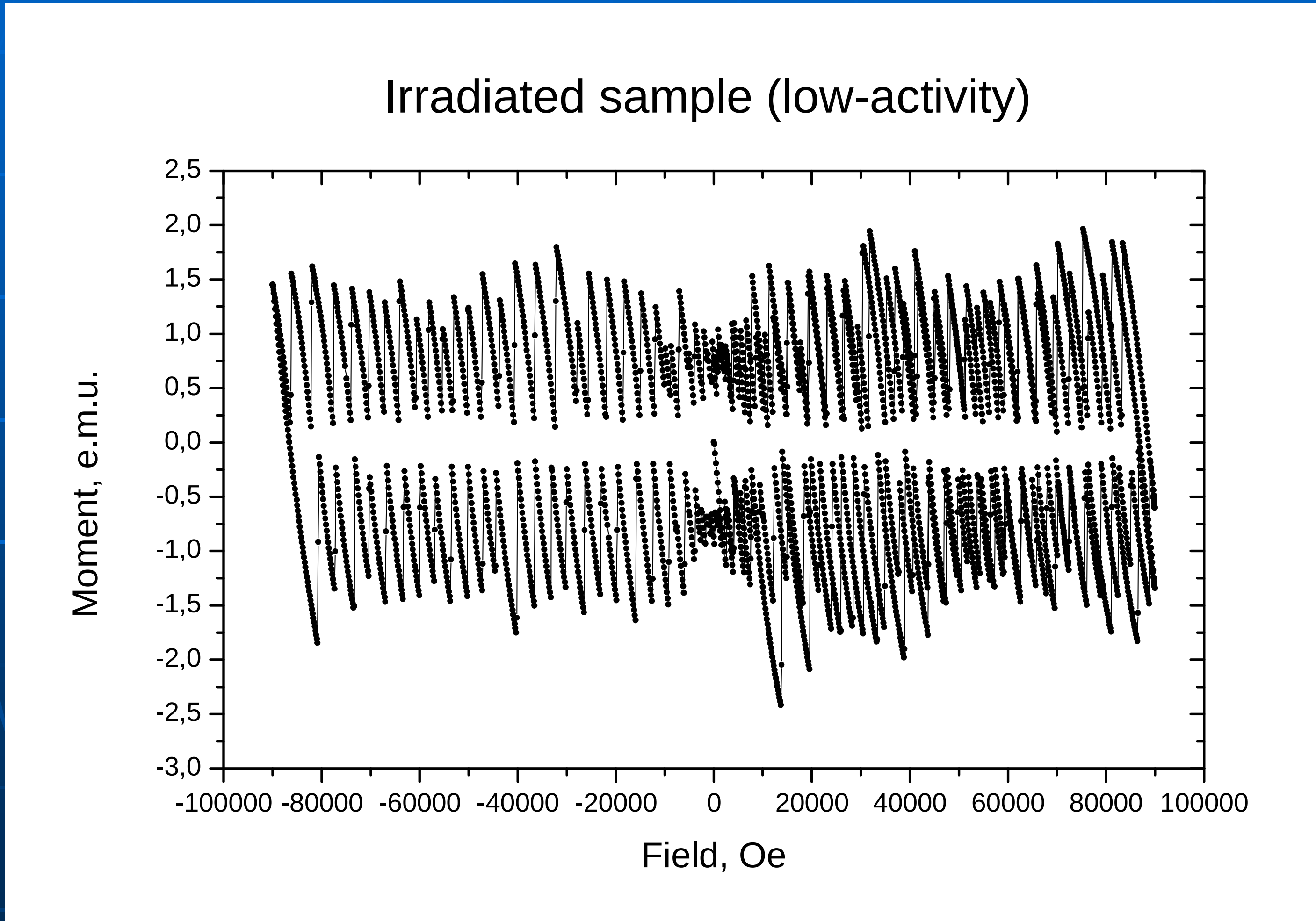
Temperature dependence of magnetic momentum



Calculated critical current density (J) vs magnetic field (H)

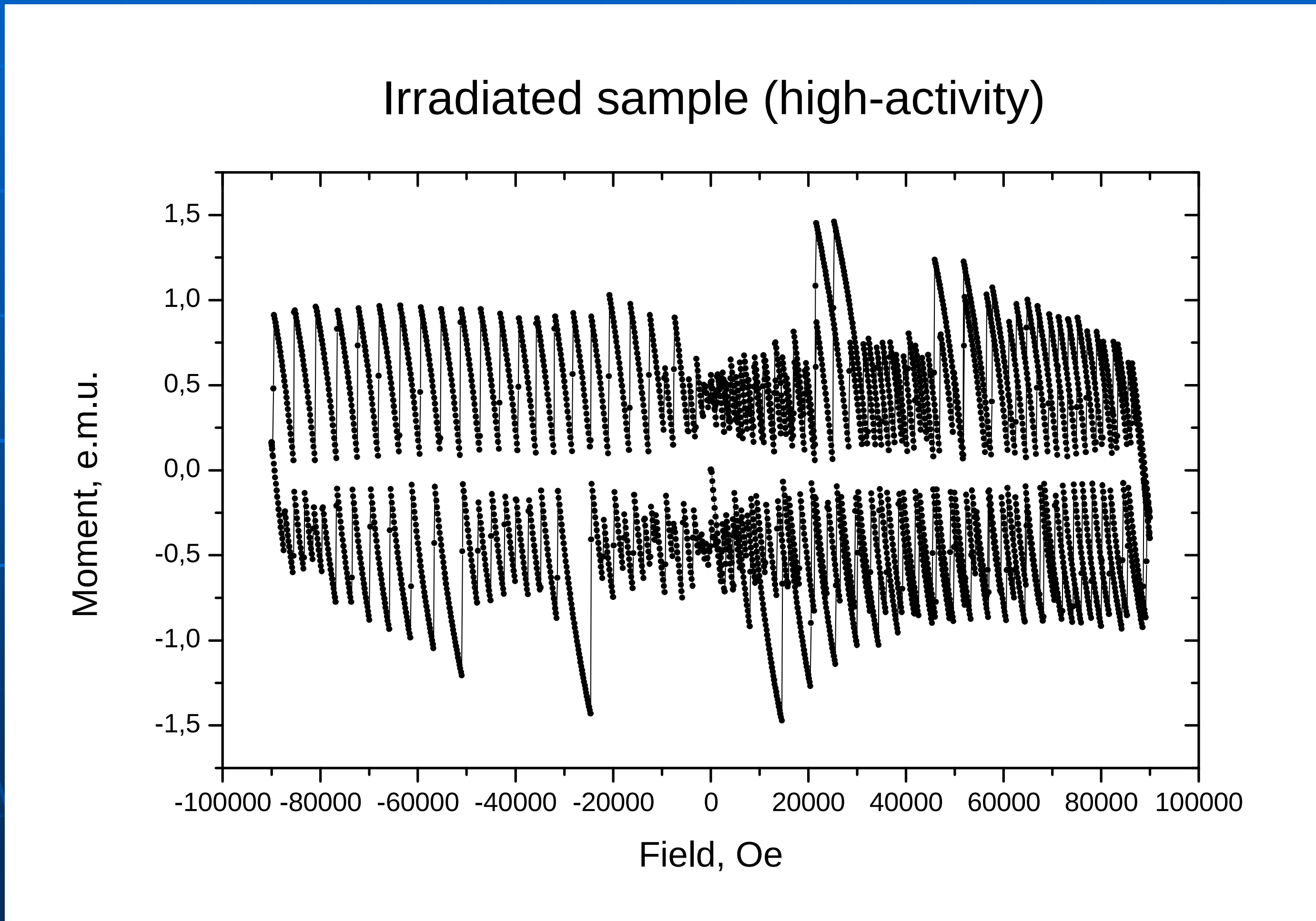


Magnetization measurements M(H) on irradiated Nb_3Sn samples by fast protons with 10 MeV energy up to total dose of 10E17 p/cm2



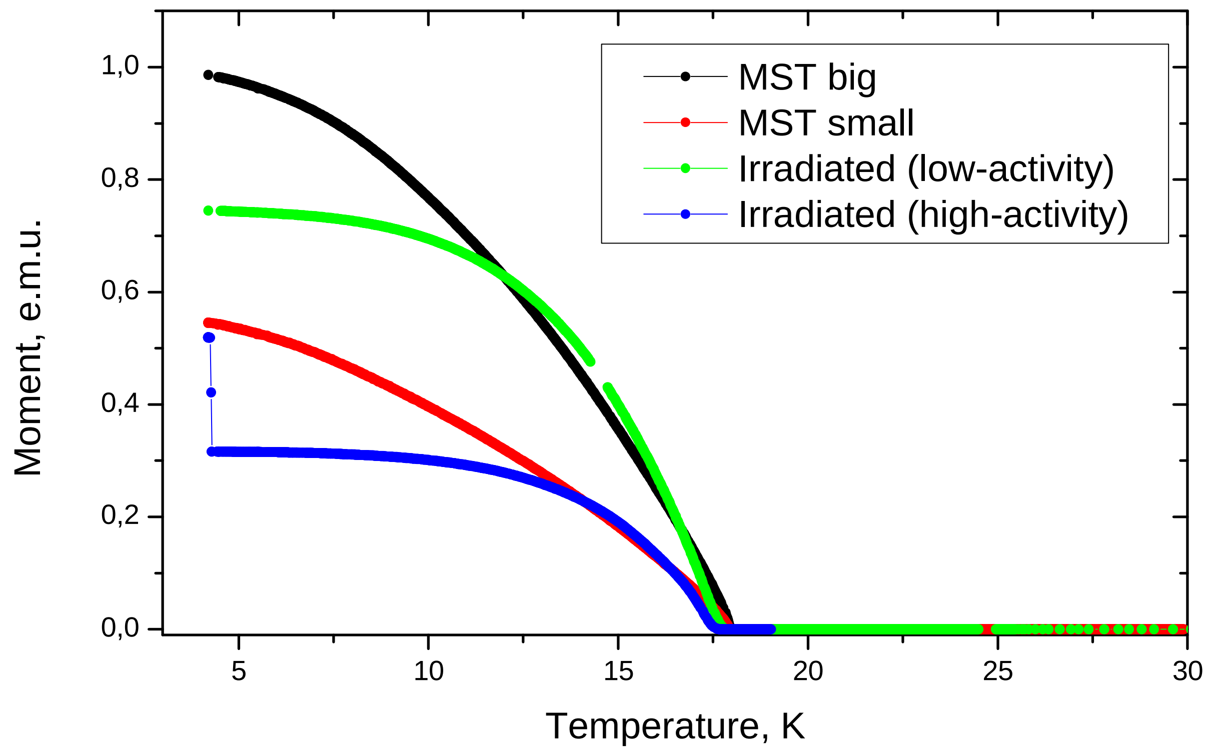
The jumps in full range occur

Magnetization measurements M(H) on irradiated Nb_3Sn samples by fast protons with 10 MeV energy up to total dose of 10E17 p/cm2



The jumps in full range occur!

Temperature dependence of magnetic momentum for unirradiated and irradiated Nb_3Sn samples



Obtained Results

- Magnetization curves were shown on Figure 1. There are some jumps in low-field region (up to 0.5 T) that can be attributed to sample inhomogeneity.

- Temperature dependences of magnetic momentum was presented on Figure 2.

- Calculated critical current density vs magnetic field up to 9 T was presented on Figure 3.

Structural study of irradiated and unirradiated Nb_3Sn samples by synchrotron X-ray diffraction

Four pristine Nb_3Sn plate samples provided by CERN have been reinvestigated using synchrotron radiation-based X-ray diffraction (##1 - 4) along with four plates irradiated by protons with 10 MeV energy on the Kurchatov cyclotron up to total dose of fast protons 10E17 p/cm2 derived from plate #2 (##2_1 - 2_4).

Sample thickness: 210 µm – 230 µm.

Penetration depth of 10 MeV protons: 285 µm

Diffraction results for pristine Nb_3Sn samples:

- All four pristine Nb_3Sn plates contain three crystalline phases:

1) in addition to the predominant phase Nb_3Sn,
2) NbO phase (Pm3m, a~4.21 Å) and
3) pure Nb phase (Im3m, a~3.31 Å).

- The exact phase composition was quantitatively analyzed within the Rietveld refinement procedure. Major crystallographic parameters for all samples are summarized in Tables 1-9. The amount of NbO in four pristine plates is about 0.3%. The maximum content of metallic Nb (2.8%) is observed for the non-stoichiometric sample #1, in other three samples this value is 0.4-0.5%.

Fig. 3. Rietveld refinement for the “as prepared” Nb_3Sn sample #2.

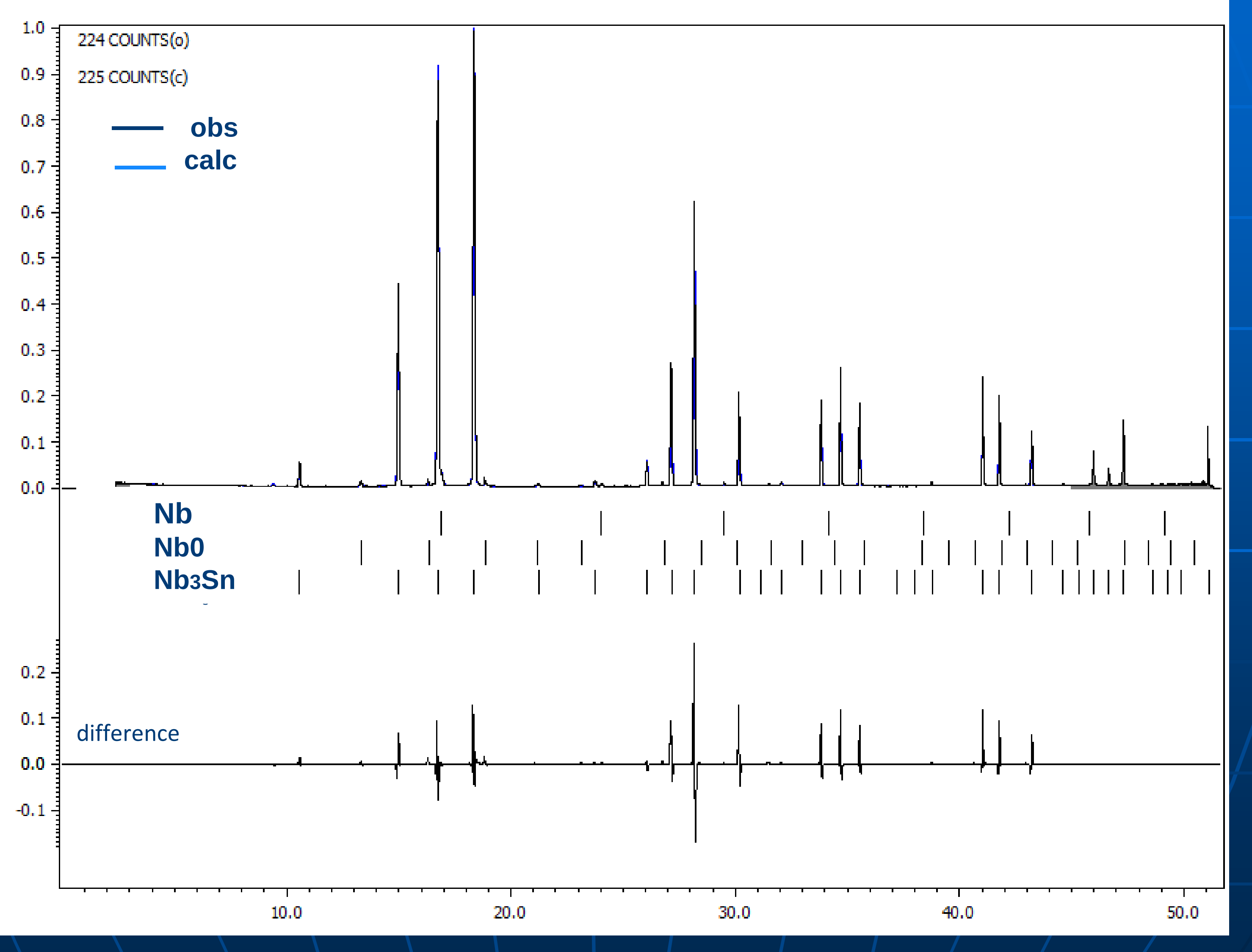


Fig. 6. Rietveld refinement for Nb_3Sn sample #2_1 after p^+ irradiation with the energy 10 MeV, dose $\Phi = 10^{17} p/cm^2$ and D = 0,007 dpa

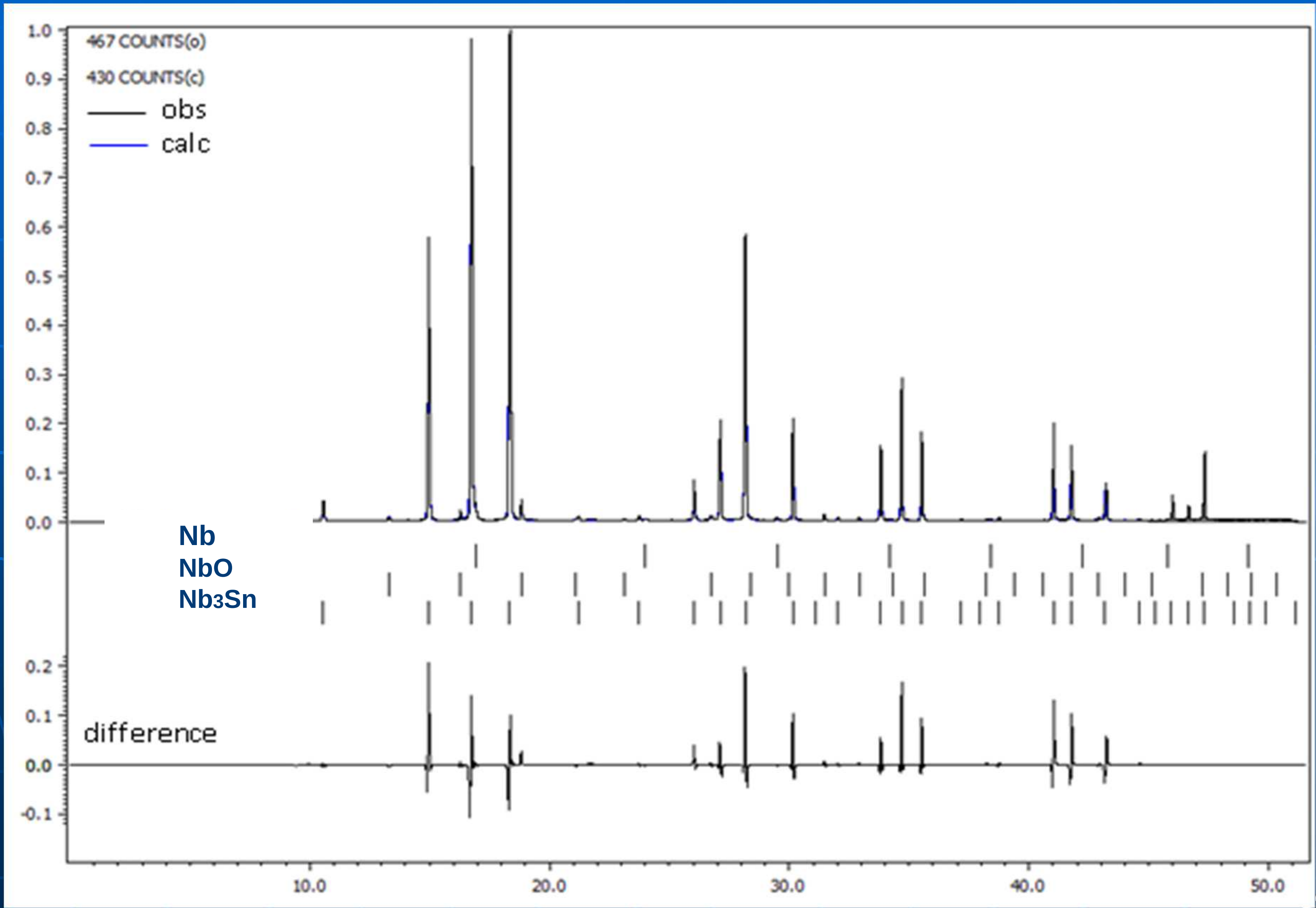


Table 3. Rietveld refinement results for initial Nb3Sn plate #2 (total Rp=15.58, for Nb_3Sn phase only Rp=5.69)

	Nb3Sn	NbO	Nb
a, Å	5.2953	4.2014	3.3147
Lx	0.5862	1.954435	5.850322
Ly	0.978505	28.35405	3.506979
shift	-1.703836	-1.703836	-1.703836
Phase,%	99.3	0.3	0.4
Uiso	Nb: 0.020585	Nb: 0.001492	Nb: 0.012809
	Sn: 0.019155	O: 0.065572	
Crystallite size, nm	673.3	201.9	67.5
microstrains,%	0.017	0.495	0.061

Table 6. Rietveld refinement results for p^+-irradiated Nb_3Sn plate #2_1 (total Rp=19.73, for Nb_3Sn phase only Rp=7.91)

	Nb3Sn	NbO	Nb
a, Å	5.2960	4.2130	3.3147
Lx	0.923881	1.093204	2.412598
Ly	2.155559	6.97876	4.593431
shift	-1.733223	-1.733223	-1.733223
Phase,%	95.4	2.7	1.9
Uiso	Nb: 0.025678	Nb: 0.003165	Nb: 0.04504
	Sn: 0.024206	O: 0.00677	
Crystallite size,nm	427.2	361.0	163.6
microstrains,%	0.038	0.122	0.080

Comparison of profiles of low-angle (left) and high-angle diffraction reflexes for pristine (#2) and proton-irradiated (#2_1) Nb_3Sn plates with the energy 10 MeV, dose $\Phi = 10^{17}$p/cm^2 and D = 0.007 dpa

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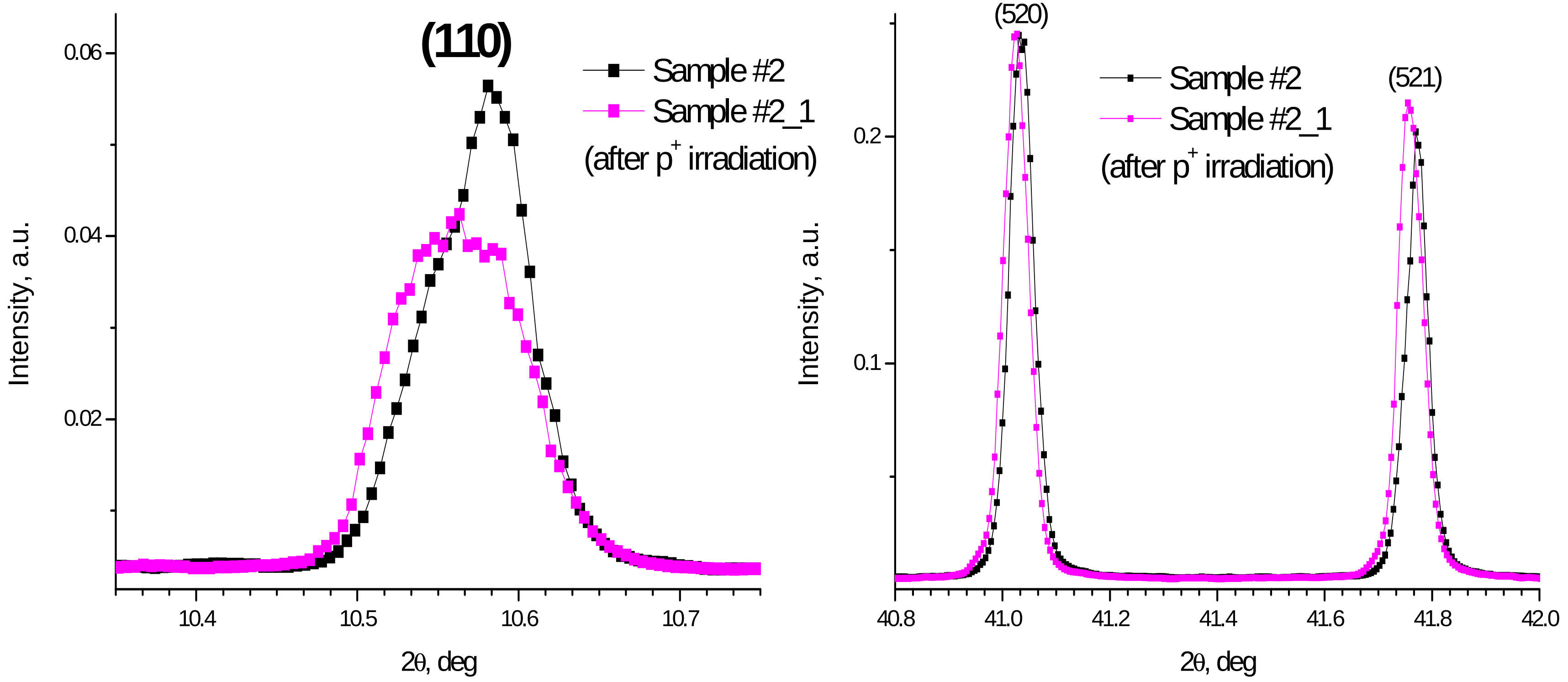
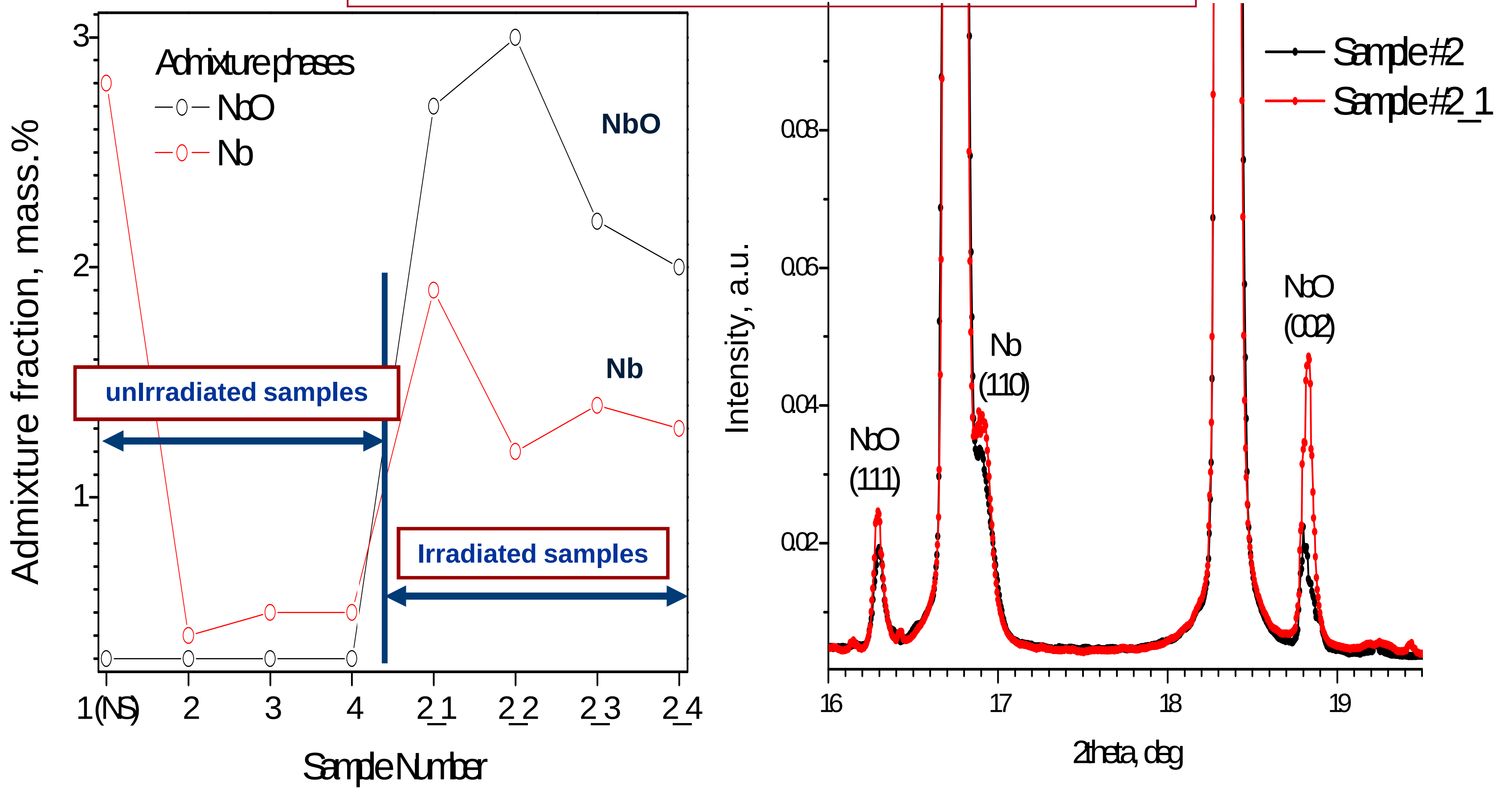


Table 10. Summary of essential structural measured parameters by synchrotron X-ray diffraction at NRC KI for irradiated by 10 MeV protons and unirradiated Nb_3Sn samples

Samples	1	2	3	4
a, Å (unirradiated)	5.2955	5.2953	5.2970	5.2983
a, Å (irradiated)	5.2960	5.2973	5.2973	5.2972
Relative intensity of the (110) reflex (unirradiated)	4.72	5.76	5.42	5.77
Relative intensity of the (110) reflex (irradiated)	4.27	5.21	4.80	4.39

Fig. 10. The fractions of admixture phases NbO and Nb in the irradiated and nonirradiated samples.

Protons with the energy 10 MeV, dose $\Phi = 10^{17}$p/cm^2 and D = 0,007 dpa



The TEM study of Nb_3Sn after the proton irradiation with 10 MeV energy

Sample preparation

Cross-sectional samples for TEM were prepared by focus ion beam (FIB) in the Helios (FEI, US) dual-beam electron-ion microscope. The Ga^{+} ions energy during sample prep was 30 keV in the beginning and 2 keV at the end of the procedure.

Tarnsmission electron microscopy/Scanning transmission electron microscopy (TEM/STEM)

TEM/STEM study were performed in TITAN 80-300 TEM/STEM (FEI, US) operated at 300 kV and equipped with Cs probe corrector system, energy dispersive X-ray spectrometer (EDXS) (EDAX, US), electron energy loss spectrometer (EELS), (Gatan. US) and high angle annular dark field (HAADF) detector (Fischione, US)

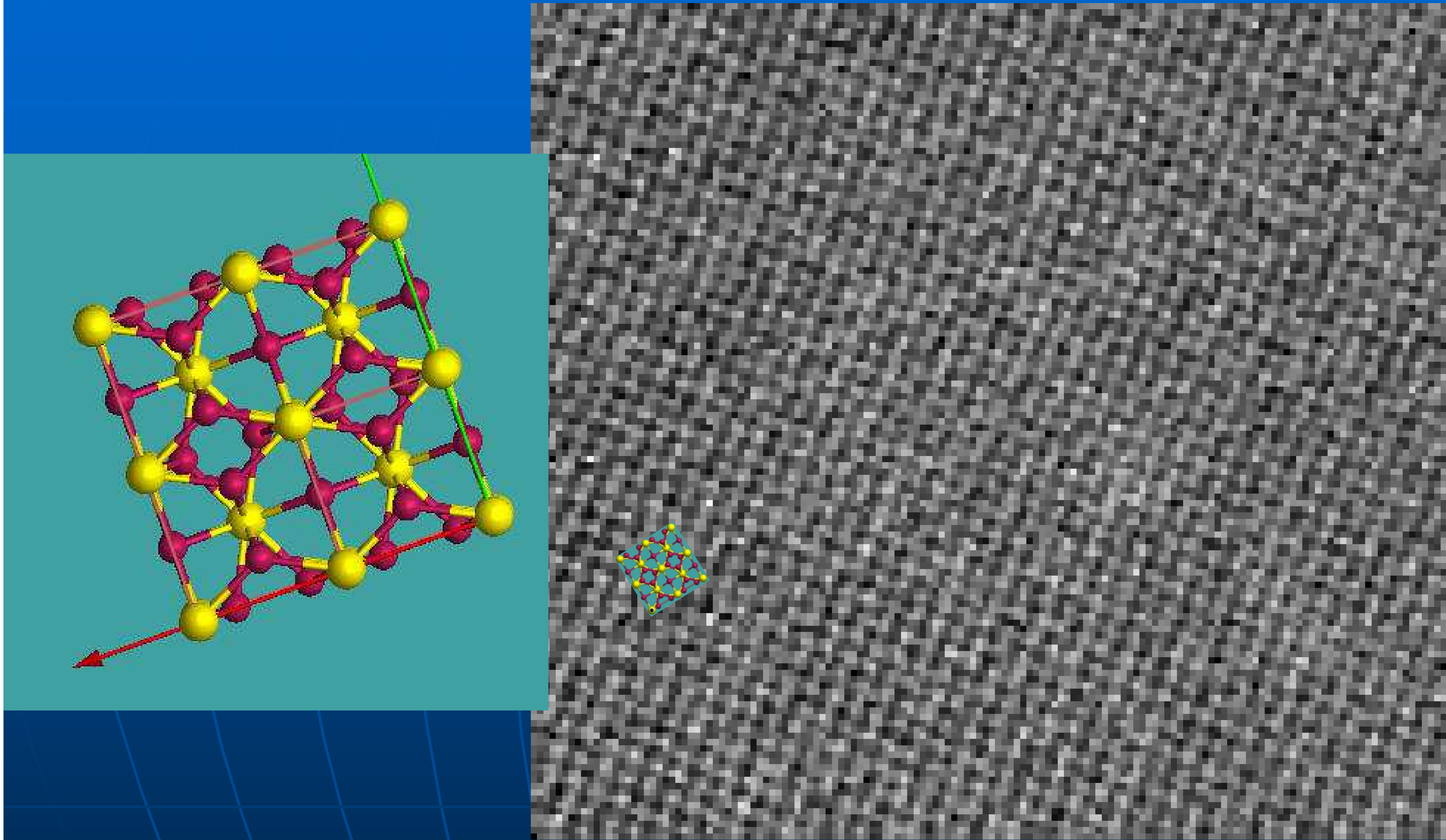
Microscopy and sample preparation

- **<u>TEM</u>:** **TITAN 80-300 (FEI, US) equipped with Cs probe-corrector (CEOS Germany), GIF (GATAN, US) and EDS (EDAX, US) at 300 kV.**

- **<u>Preparation technique for samples.</u>**
  2 techniques were explored:

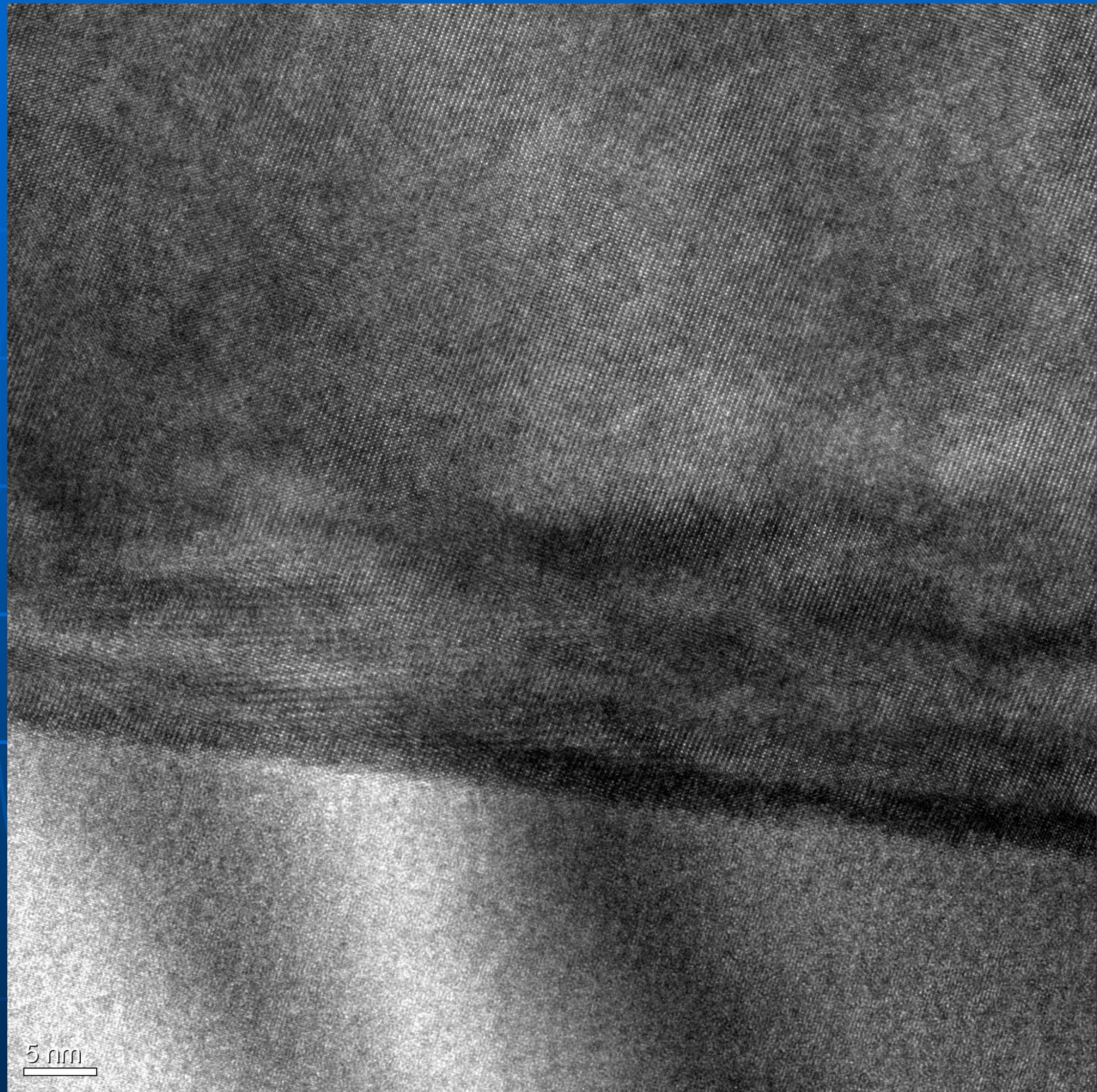
- **A. Mechanical thinning and polishing to 50-70 µm followed by Ar^{+} milling in GATAN PIPS (GATAN, US) at 5 kV with 0.2 kV in final step.**
- **B. FIB etching in HELIOS (FEI, US) with Ga^{+} at 30 kV followed by 2 kV at final step.**

Nb_3Sn sample #22 after ion milling



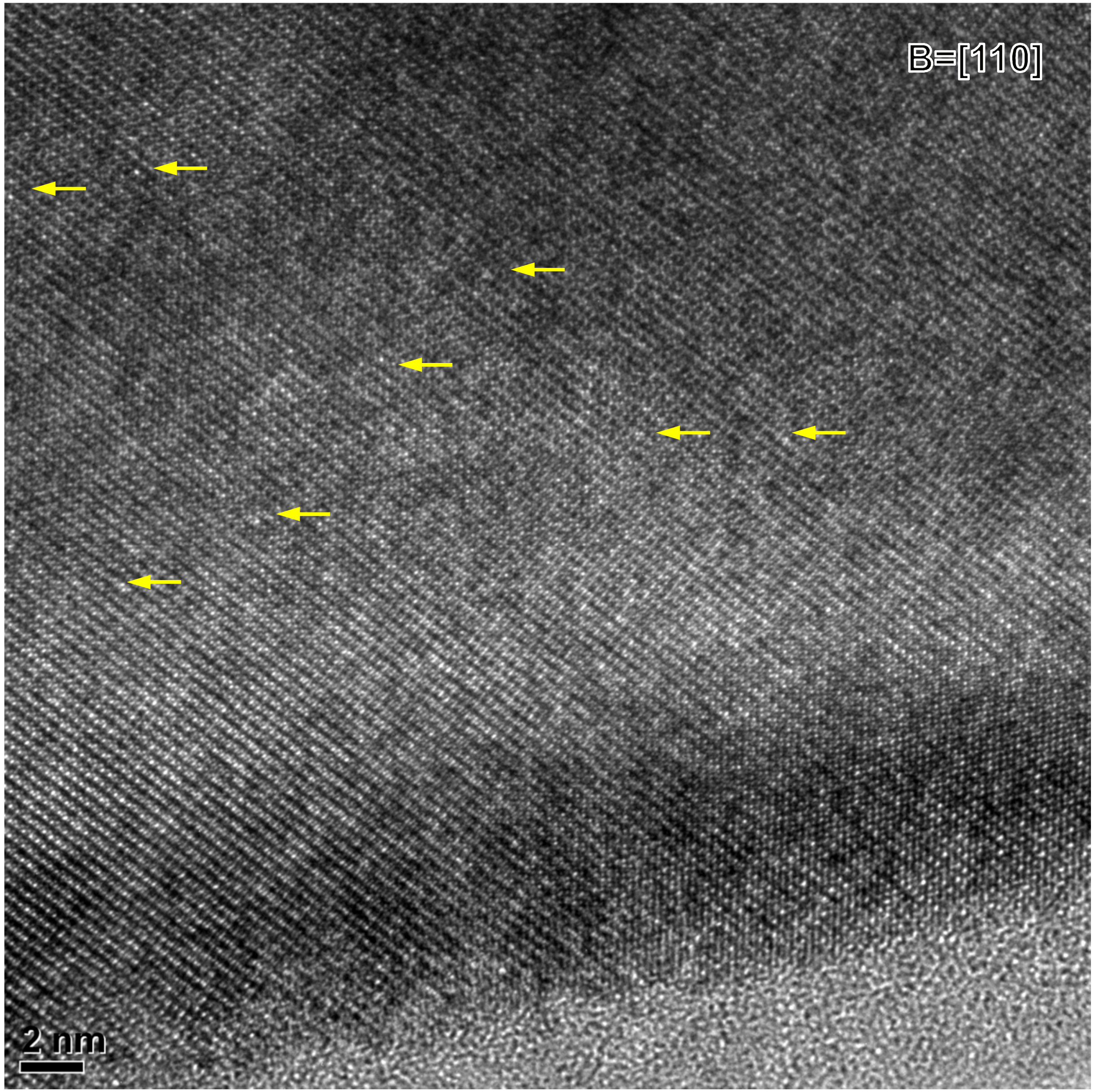
High resolution scanning transmission electron microscopy with high angle annular dark field image (HR STEM HAADF)

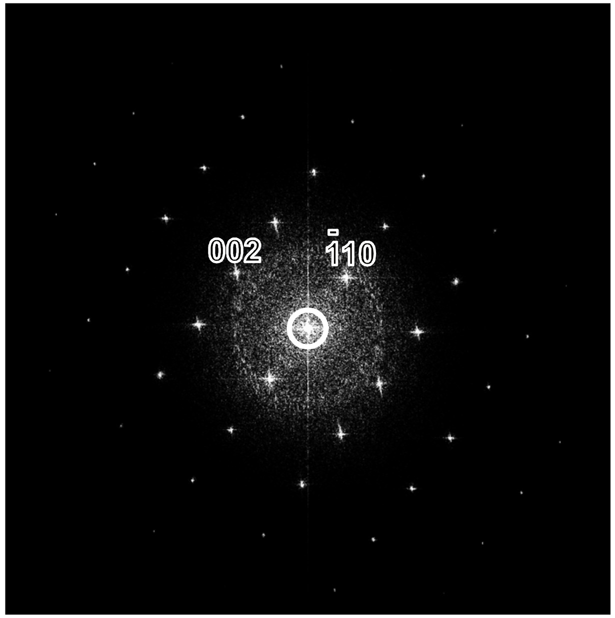
Nb_3Sn sample #22 after FIB



Bright field (BF) HR TEM image of the grain boundary (GB).

The bright field (BF) high resolution TEM (HREM) image and Fast Fourier Transform from the image after p - irradiation

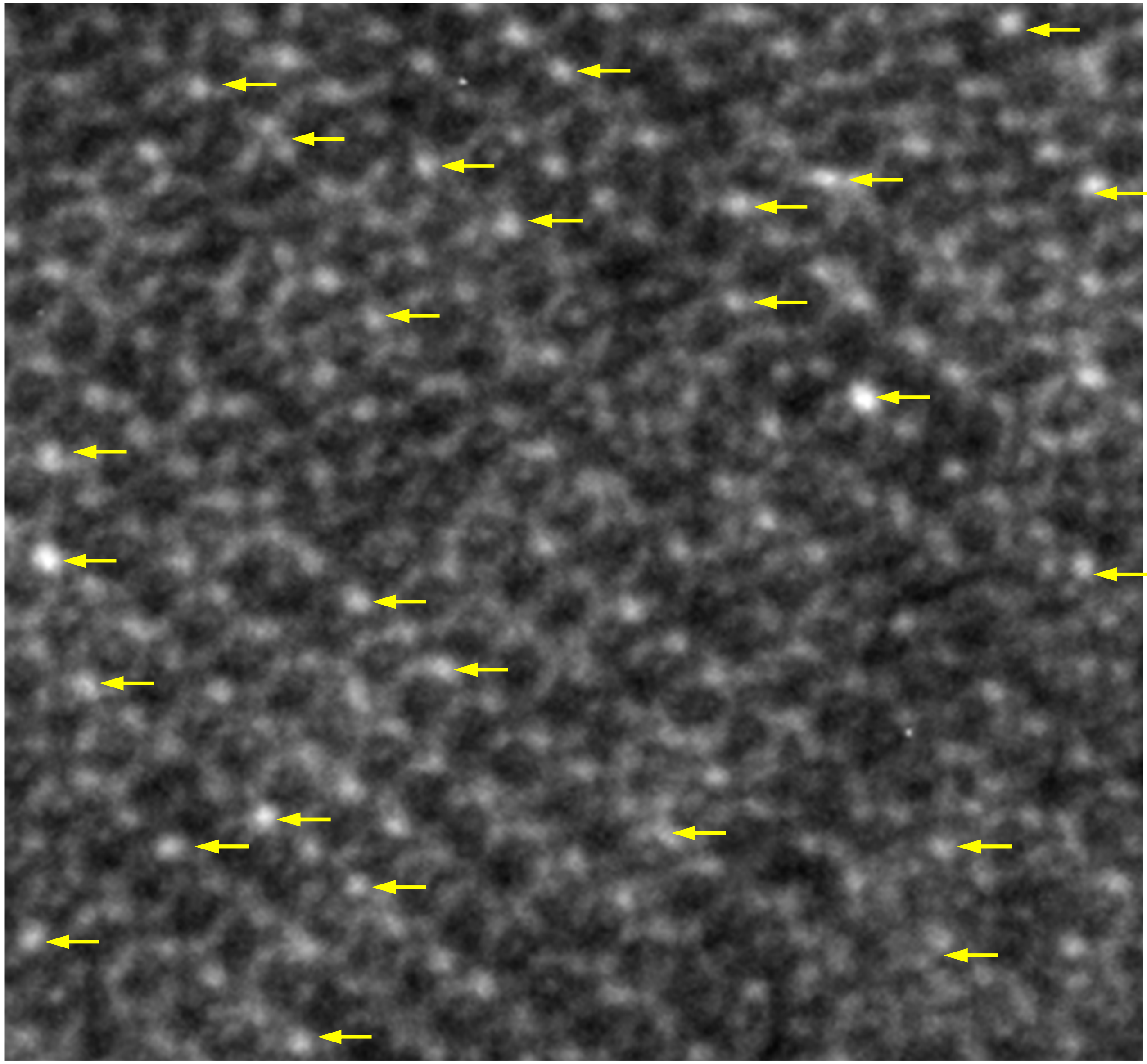




Local contrast deviations which could arise due to the radiation vacancies in the crystal lattice are shown by arrows. Few of them were observed on several images obtained with time interval of 30 seconds.

Close inspections of the micrographs demonstrated the 3-5 % increasing of the unit cell parameters in Nb_3Sn

Enlarged BF HREM image after proton irradiation



Note, that the radiation defects are located in the same positions. More definite answer on the contrast variations and possible influence of vacancies on HREM image will be obtained after image simulations.

Obtained Results:

- Nb_3Sn particles were 1-5 µm in size and irregular in shape. Preliminary results obtained on unirradiated samples demonstrated the stoichiometry of the particles were 18-26 at % Sn and 74-82 at % Nb, however few particles were found with 28 at % of Sn. The Sn deficiency cause the monoclinic distortion of A15 lattice. The grain boundaries were free of intermediate layers. The dislocation density was very low we failed to reveal any dislocations within the grains.

- The radiation defects have been observed in irradiated Nb_3Sn samples on NRC KI cyclotron by fast protons with 10 MeV energy, dose Φ = 1017p/cm2 and D = 0,007 dpa.

- These defects are located in some positions. More definite answer on the contrast variations and possible influence of radiation vacancies on HREM image will be obtained after image simulations.

- The results of X-ray diffraction obtained on Synchrotron Source at NRC KI revealed the widening and intensity decrease of diffraction peaks as concentration of radiation defects increasing.
- New phases: NbO and Nb were founded in the irradiated material. The density of these phases was growing with the increasing accumulation of radiation defects.
- Research was also carried out on changes in the lattice parameter which was increased with increasing of irradiation dose.

Thank you very much for your attention!